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MARCH 27, 1975 - DECEMBER 31, 1976

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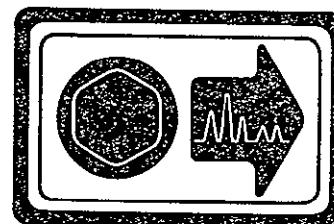
DEVELOPMENT OF WATER QUALITY STANDARDS CRITERIA

TECHNICAL MONITOR: R. L. SAUER

TO

THE NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
JOHNSON SPACE CENTER
HOUSTON, TEXAS

DECEMBER, 1976



**SPECTRIX
CORPORATION**

FINAL REPORT

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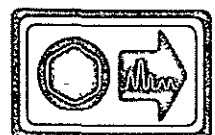
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TABLE OF CONTENTS

	Page
1.0 INTRODUCTION	1
2.0 OBJECTIVES	3
3.0 EVALUATION OF TASTE ENHANCEMENT FACTORS	3
3.1 Experimental Approach	3
3.2 Results and Discussion	5
4.0 ANALYSIS OF VOLATILE ORGANICS IN VCD SAMPLES	8
4.1 Description of Samples	8
4.2 Analytical Procedure	10
4.3 Results and Discussion	12
5.0 INFLIGHT ADDITION OF TASTE ENHANCERS	16
6.0 CONCLUSIONS AND RECOMMENDATIONS	17
6.1 Spacecraft Water Quality Potability Specifications	17
6.2 Further Evaluation of Reuse Water Systems	18
7.0 SUMMARY	19
8.0 APPENDICES	
8.1 Appendix I: Tables	
8.2 Appendix II: Figures	

1.0 INTRODUCTION

The requirement to supply metabolic and personal hygiene water onboard future spacecraft necessitates water reclamation from urine, feces, and wash water. Systems are currently being built and tested to provide this reclamation and thereby close the water loop. The use, and in particular, the human consumption of this reclaimed water raises questions as to its continued potability and palatability. Because of the nature of the source of the reclaimed water and the possibility of the carry over (during the reclamation) and/or selective concentration of specific potentially toxic organic materials, it is imperative that the organic constituents of the water be precisely determined. This is necessary to determine and verify the absence of potentially toxic and adverse taste producing compounds.

The removal of essentially all inorganic constituents by the reclamation process produces water characterized by an unpalatable flat or bitter taste because of the lack of specific ingredients. Determination of the minimum ingredients required and readdition of these essential ingredients prior to consumption will enhance the taste of spacecraft water supplies to the level required to encour-

age space travelers to consume sufficient water to meet their physiological needs.

Under previous contractual effort, a technique employing an absorbent resin coupled with gas chromatographic-mass spectographic analysis was developed. This analytical procedure supports qualitative analysis of the volatile constituents in water and was applied to the analysis of urine feedstock and product water from some early prototype water reclamation systems. In addition, preliminary efforts were successful in applying this technique for quantitative analysis. This earlier work also resulted in the development of preliminary criteria and specifications for potable/palatable water to serve as guidelines for subsequent phases of investigation. A preliminary parametric investigation of palatability factors was conducted with the goal of determining the optimum addition of specific chemical ingredients to maximize taste enhancement. The results of the organic analyses of the water samples indicated potentially toxic substances in the product water and pointed to the need for careful sampling and analysis for evaluation of future systems. The palatability investigations indicated that no organic compounds need be added to ultrapure water to enhance taste and that perhaps only a few inorganic species

were actually required to produce the desired palatability effects. The minimum number and quantities of ingredients was yet to be determined.

2.0 OBJECTIVES

The principal objectives of this study effort have been (1) to perform qualitative and semi-quantitative analysis of volatile organic compounds in water samples collected at various stages of processing in the most advanced reclamation system now being developed within the NASA, the Space Station Prototype Vacuum Compression Distillation (VCD) unit, for the purpose of evaluating the process and the product water; (2) to perform additional evaluation of the specific ingredients required to adequately enhance the taste of reclaimed water; (3) to develop a preliminary concept for the inflight addition of these ingredients; and (4) to make any revisions to previously recommended potable water criteria and specifications deemed necessary as a result of this study.

3.0 EVALUATION OF TASTE ENHANCEMENT FACTORS

3.1 Experimental Approach

Table 1 gives the factors known to be related to the palatability of water. It was not known what the minimum number

and amount of chemical constituents must be added to an ultrapure (no detectable inorganic or organic constituents) water to produce an acceptable testing water. Previous taste panel evaluations (Final Report: Potable Water Taste Enhancement, Contract NAS9-12969) indicate that Ca^{++} , K^+ , Mg^{++} and Na^+ added to the ultrapure water produces a significant improvement in taste. We have performed additional taste evaluations to better define the minimum amounts required for maximum palatability and to determine whether all four cations are essential. The effect of chloride and sulfate anions in conjunction with the added cations was also investigated.

Ultrapure water was prepared from deionized water by distillation with potassium permanganate in a glass fractionating column. Atomic adsorption analyses (Water Analyses by Atomic Adsorption Spectroscopy, by C. R. Parker, Varian Techtron Pty. Ltd., Springvale, Australia, 1970) was used to verify that inorganic cation contents were less than 0.3 ppm. Total particulate and organic carbon contents were verified to be less than 1 ppm by a total carbon combustion analyzer utilizing an infrared detector. Headspace extractable organic volatiles was verified to be less than 1 ppb by the adsorbent trapping and analysis

method described later. Samples for taste evaluation were synthesized by adding the appropriate chemical constituents to this ultraclean water according to the matrix given in Table 2. This matrix was developed with the following objectives in mind: (1) to determine if both Cl^- and SO_4^{2-} are desirable, (2) to determine if Ca^{++} , Mg^{++} , K^+ and Na^+ are all required and (3) to determine the upper allowable limit for cations and anions. Sample 1 has the concentrations selected from the results of previous taste tests (ibid). Dissolved oxygen content of all samples was 8.0 ppm and dissolved CO_2 was the value for equilibrium with air (5 ppm).

3.2 Results and Discussion

The synthesized water samples were evaluated by a taste panel comprised of five members. The members ranged in age from 24-47 years. One member was a female. Three of the members had previous taste panel experience. All were professional chemists. Samples were tasted at 72° F, since Pangborn and Bertolero (Journal AWWA, pp. 511-515, August, 1972) have shown this to be the temperature at which the intensity of taste is greatest, and compared to the ultrapure water. The data for the first set of samples which is given in Table 3 was obtained by pairing the synthetic sample with the ultrapure blank. Both the ultrapure "reference" and the sample were 15 ml volumes in

50 ml acid cleaned beakers. The tasters were instructed to fill their mouths to whatever volume is comfortable, hold it for several seconds, and discharge it without swallowing. Tasters were asked to grade the water samples on a scale of 1 - 10 with the ultrapure "reference" sample defined as five. Average grades and standard deviations are given in Table 3. Tasters were not provided any information on sample water composition prior to tasting trials.

A second set of taste tests was then conducted with the same water samples using a different evaluation scale but with all other parameters identical to the first tests. The results of these tests are given in Table 4, Part A. Tasters were asked to rate the samples on a Hedonic scale from 0-10 with 0 being very bad tasting and 10 being very good tasting. An acceptable sample was given a numerical rating of 5. Tasters were also asked to rate the samples after swallowing since this is the normal situation when drinking liquids. These results are given in Part B of Table 4.

Figure 1 gives the relative positions of the various samples tasted. It can be seen that Sample #2 containing all sulfate anions is definitely rated low in both tests. The ultrapure

water sample was given a rating slightly less than acceptable on the Hedonic scale indicating that it would not be objectionable. The samples containing added cations and Cl^- anion are generally rated higher than the ultrapure water in both tests. It also appears that both Na^+ and K^+ are not essential, but that one of these two cations is desirable. It should be noted that samples containing only CaCl_2 and NaCl_2 received as good a rating as any samples tested.

The tasters were asked to swallow the water and record their evaluation in the second series of tests to determine if this would substantially change the ratings. Taste procedures described in the literature are designed for threshold detection of bad taste. We felt that swallowing the water as one would in normal drinking would be a more valid test for determining good tasting water. It can be seen in Table 4 that swallowing has very little effect on the ratings.

The results of these tests indicate that addition of only one salt, either CaCl_2 or NaCl_2 may be sufficient to the desired taste enhancement. We do not feel this can be conclusively demonstrated, however, until a large taste panel (20 or more persons) is used for testing and which is beyond the scope of this work. Even then it may be desirable to obtain taste re-

actions from the actual spacecraft crew to determine final species and concentrations.

4.0 ANALYSIS OF VOLATILE ORGANICS IN VCD SAMPLES

4.1 Description of Samples

A basic understanding of the operation of the VCD is required for understanding the nature of the samples which were analyzed in this study. Figure 2 is a schematic diagram of the VCD module (Chemtrac Report 3110, Contracts NAS 9-13714 and NAS 9-14234:Chemtrac, Inc., 9330 West William St., Rosemont, Illinois 60018, March, 1975). Operational requirements for the module are divided into five categories including (1) waste water storage to receive and contain pre-treated urine, (2) a distillation unit for initial purification, (3) a distillate post-treatment unit for final purification, (4) an automatic control system and (5) modular packaging for inflight maintenance. Only the distillation and post-treatment units are of importance to this study so further description will be limited to these subsystems.

Three liquid streams cross the distillation unit boundary and interface with the remainder of the VCD module - namely, (1) waste liquid entering the evaporator, (2) waste liquid leaving the evaporator, and (3) distilled water leaving the condensor. These streams are referred to as the feed, recycle, and condensate, respectively. The feed and recycle streams are portions of a recirculating waste-liquid loop

which passes between the recycle tank and the evaporator. A portion of the water fed to the evaporator is removed as it passes through the evaporator and this extracted water becomes the condensate stream. The volume of water extracted from the loop is replaced by a like volume of waste delivered by the waste tank. Noncondensable gases are removed from the evaporator by a purge pump. The condensate stream is passed through the post treatment unit where a biological filter removes any bacteria, a charcoal filter removes any co-distilled organic contaminants and Ag^+ ions are added as a sterilant before going to a storage tank contained external to the VCD module.

Five samples were provided by the VCD laboratory for evaluation in this study. These samples were acquired during operation of the VCD prototype in the summer of 1976. Sample names and descriptions are as follows:

Urine + Pretreat	Taken from the feed used to charge the waste storage tank. Consists of urine and a pretreat mixture containing an iodoform and antifoam agents.
Recycle Fluid	Taken from the recycle tank (see Figure 2).
Untreated Product	Taken from the condensate line (see Figure 2) prior to any post treatment.
Product Water	Taken from the condensate line (see Figure 2) after charcoal filtering.
Distilled Water	VCD laboratory distilled water control

4.2 Analytical Procedure

Volatile organic compounds in the samples were concentrated on a solid adsorbent trap by a headspace sampling technique. Figure 3 shows a cross sectional view of the trap and the modified gas chromatograph injector port which is used to desorb the trap. The adsorbent was Tenax GC 35/60 mesh (supplied by Applied Sciences, Inc., State College, PA). Figure 4 shows a diagram of the sampling apparatus. The sample liquid is placed in the stripping flask and heated to 100°C. Ammonium sulfate (30% w/v) was added to enhance volatilization. Sample volumes were 50 ml with the exception of the recycle fluid where only 15 ml was available. After the sample was heated the volatiles were stripped by bubbling helium through the sample and then onto the Tenax trap at a flow rate of 20 ml/min for one hour. The stripping gas and volatiles are passed through the water condensor prior to introduction to the adsorbent trap to minimize water content in the trap. The Tenax trap was then stored in a clean teflon lined screw cap sealed Pyrex vial for subsequent analysis.

The analyses were begun by removing the trap from the storage tube, inserting it into the modified injector port, and sealing the port. Figure 5 is a schematic diagram of the sample transfer and analysis system. The volatile organics were thermally desorbed at 225°C for 20 minutes into a liquid nitrogen cooled capillary precolumn (10' x 0.02" i.d.). After the allotted transfer time the coolant was removed and

the precolumn rapidly heated to 250°C. to produce an injection onto a 200' x .02" i.d. nickel capillary column coated with DC-200 silicone oil. The multiport valve allows switching of the carrier from the desorption-vent to the inject-analysis position without interrupting GC column flow.

A Finnigan 3200-Incos 2300 gas chromatograph-mass spectrometer data system (GC-MS-DS) was used for the analysis of the volatiles. Temperature programming was used $[30^{\circ}(12 \text{ min}) \xrightarrow{2^{\circ}/\text{min}} 180^{\circ}]$ to produce better separation. The GC column was interfaced to the mass spectrometer through an all glass jet separator maintained at 250°C. Mass spectra were recorded every three seconds by the data system throughout the entire chromatogram and stored on a magnetic disk. Background subtraction (enhancement) was automatically performed by the data system to remove column bleed and other instrument background contamination. The resultant enhanced spectra were library searched by the data system against the 19,000 spectra NIH/EPA library maintained within the data system. All identifications produced by library search were verified or rejected by manual inspection. All remaining unidentified peaks were identified by manual procedures, if possible, using the Aldermaston eight peak index and knowledge of fragmentation procedures.

Estimates of the quantities of the various components present were made by calibrating the GC-MS-DS response with known amounts of tetradecane. Areas of the standards and the sample

components were compared to obtain estimates of sample component quantities. All volatiles are reported as tetradecane and it should be noted that these estimates are subject to considerable error since the compounds could have responses differing by the ratio of as much as 5:1 and may not be extracted with equal efficiency due to concentration or chemical differences.

4.3 Results and Discussion

Figures 6 through 10 are the reconstructed gas chromatograms (RGC) for the five samples analyzed. The RGC's are the total summed resolved ions currents for each mass spectrum (total ionization) plotted for each scan number and normalized to the most intense scan in the chromatogram. Figures 11 through 15 show these same RGC's with the horizontal time scale expanded to 500 scans per page and the total ionization per scan plotted for three different vertical scales (1:10:100) so that very small peaks can be seen. Tables 5 through 9 list all compounds identified in the various samples, their formulas and molecular weights, the estimated amounts trapped on the Tenax, and the weight percent of each component. The total estimated volatiles (ppb in wt/wt) are also given at the end of the listings.

Table 10 gives a comparison of the concentration of all the identified compounds in the four samples from the VCD

module. The principal compounds found in the urine + pre-treat sample are the iodides and dioxane from the iodoform plus the siloxane compounds used as the antifoaming agent. The sulfur compounds, aromatics, alcohols, ketones, aldehydes and paraffins have previously been reported in urine samples (Final Report NAS9-12093, University of Houston, 1975) and are not surprising. The absence of pyrroles, pyridines and other nitrogen containing compounds is somewhat surprising but urine samples are known to vary widely in composition even from the same subject.

The principal constituents of the recycle fluid are the dioxanes, the C_5 alcohols and the siloxanes. The recycle fluid should be a concentrate of the less volatile components, the amount of concentration depending on the length of time the module has been in operation. The compounds are listed in Table 10 in order of their elution on the nonpolar DC 200 column and should, therefore, also be in the order of decreasing volatility. A nominal operating distillation procedure should result in increasing ratios when comparing the recycle fluid to the urine + pretreat proceeding through the list. This is generally the case since the early volatiles are not present in the recycle fluid. The siloxanes and the more abundant urine constituents tend to show less concentration. This may be due to saturation of the Tenax adsorbent because of the larger quantities involved and/or to sporadic operation of the VCD module.

The untreated (condensate) product contains three components (dioxane, a C₅ alcohol and menthol) which account for nearly 90 percent of the total volatiles. Ethyl sec-butyl ether, propyl alcohol, 2-ethyl hexanol, and 4-tertbutyl cyclohexanone and menthol were found in this sample and not in the urine + pretreat-feed or recycle fluid samples. Except for the menthol these compounds were probably not detected in the urine + pretreat and recycle fluid because of interferences from very large neighboring peaks in the chromatogram. The presence of menthol may be due to secondary reactions or a contaminant introduced by a smoker sometime during operation of the module or sample collection. The dioxane, C₅ alcohol(s), siloxanes, and other compounds common to the three samples are evidently carried over from the distillation. A crude estimate of the toxicity of the 21 compounds identified in this sample was obtained by looking them up in the Merck Index (Merck & Co. Inc., Rahway, N. J., 1968). Dimethyl styrene, the siloxanes, dimethyl disulfide and the two paraffins were not listed. Ten of the compounds are classified as toxic to some extent with dioxane being potentially the most toxic. The estimated total volatile organic content of the untreated condensate is 1310 µg/L compared to 5070 µg/L and 7100 µg/L for the urine + pretreat and the recycle fluid, respectively, indicating that the distillation is removing about 75 percent of the volatiles in the feedstock. This would not be considered a good laboratory distillation but laboratory distillations usually are carried out from an alkaline permanganate solution in order to oxidize organic material.

Also, organic content of the feedstock is usually less than 1 ppm in our laboratory distillation. It should also be noted that the bulk of the volatile organics in the untreated product water sample are in the mid and high boiling range (100 - 200° C). Normally one would expect higher concentrations of the more volatile or low boiling components in a condensate, but these are probably removed by the purge pump.

The distilled water was obtained from a Super Q filtration apparatus and was supplied as a control. Only two volatile organic components (see Table 8) benzene and acetone, were found in this sample and the total volatiles were 166 µg/L which is considerably cleaner than the two condensate samples.

The total estimated volatiles in the post treated product water is 1280 µg/L and is equal to that of the untreated condensate. The distribution of the individual components, however, is very different than in the untreated condensate (see Table 9). Acetone, methylene chloride, ethyl alcohol and benzene make up 99.8 percent of this total. We can offer no definitive explanation for the much higher concentrations of the four components mentioned above as compared to the untreated condensate. We would speculate, however, that these components may have been used as cleaning solvents during assembly of the apparatus and may be picked up by the water after it passes through the charcoal filter. There are ten components in this sample which could not be identified

because of their low concentrations - all are in the 0.1 to 0.5 $\mu\text{g/L}$ concentration range. These unidentified components are most likely the same compounds as are present in the untreated condensate. It would be very instructive to conduct a true closed loop recycling test with product water being consumed and the urine being returned as feedstock to determine if these components gradually build up in concentration over extended periods of time.

The estimated total volatile organic contents of the product water sample analyzed was 1.3 mg/L and deserves some comment since total organic carbon (TOC) for this sample was 30 mg/L (private communication from C. Verotsko, NASA-JSC, Houston, Texas). Several factors can be all or in part responsible for this apparent discrepancy. First, the average carbon number of the product water is 5. 30 mg/L of TOC is therefore equivalent to 6 mg/L if due entirely to volatiles. Secondly, the TOC may partly be made up of high molecular weight organic compounds which are not removed by inert gas stripping. Thirdly, the estimates of quantities may be off by factors of 2-5 as discussed earlier, since quantitation is based on the GC-MS response for tetradecane. Finally, very minute particles of charcoal may have been picked up from the filter.

5.0 INFLIGHT ADDITION OF TASTE ENHANCERS

Providing water that will have a good enough taste to encourage the space traveler to consume sufficient quantities to meet

basic physiological needs will definitely require the addition of some chemical species to the recycled product water. Taste tests performed indicate that the capability to add sodium, magnesium, potassium, and calcium as chlorides or nitrates will be sufficient to provide suitable taste enhancement. Concentrations required will be in the 2-20 ppm range. This addition can be accomplished simply by maintaining a concentrated solution on board the spacecraft and adding it to the water supply. Miniature valving systems similar to those used on automatic laboratory analyzers and the Viking biology experiment would inject small amounts of the concentrate into a dilution-holding chamber located just prior to a drinking water outlet. (The addition system must be placed after the deionizer which removes the Ag^+ biocide present in the main product water storage tank). The dilution-holding tank could be sized to hold a one day supply of drinking water and may even be cooled to further enhance palatability. One of the cations can be monitored by a specific ion electrode for quality control purposes if desired.

6.0 CONCLUSIONS AND RECOMMENDATIONS

6.1 Spacecraft Water Quality-Potability Specifications

Techniques developed in this and earlier study efforts have permitted the qualitative and semi-quantitative analysis of volatile constituents in product water obtained from a prototype water reclamation unit. These results indicate the presence of potentially toxic levels of specific organic

contaminants in the final product water even though the reclaimed water meets the total organic requirement of existing terrestrial specifications for potable water. This is not to say that these specifications are inadequate for their intended use, but only that they must be expanded, particularly in the area of organics, when applied to evaluating reclaimed water. The formulation of USPHS and other similar terrestrial water specifications has been a complex undertaking involving many scientific disciplines. In addition, and most importantly, these specifications evolved over many years of real use experience in which it was determined that waters containing less than the specified amounts of a particular constituent were acceptable for human consumption. However, these criteria do not consider the peculiar problems associated with the reuse of recycled water. We recommend that a study effort be undertaken to define a protocol by which comprehensive reclaimed water potability/palatability criteria can be established and updated.

6.2 Further Evaluation of Reuse Water Systems

The results of this study indicate that 10-12 chemicals of potential toxic nature may be present in the prototype VCD product water at concentrations of 0.1 ppb or greater. Additional samples should be collected during subsequent operation of the VCD module for further evaluation. More sensitive methods of analysis should be applied in order to identify

more of these components. This can be done by stripping larger volumes of sample and by a data processing technique known as mass chromatogram plotting. The data processing involves searching sets of three or four of the most intense ions in the spectrum of the compound of interest over the retention time range where it is expected to elute in the chromatogram. Simultaneous maximization of these ions indicates the presence of the compound. Compounds identified in the feed or recycle samples can be searched in the product waters in this manner.

The question of the buildup of toxic chemicals in a closed loop recycling system should be addressed. Samples analyzed to date and planned for the next study are not representative of a closed cycle. Samples of product water should be obtained at regular intervals over a 30 - 60 day operating period when the product water is being consumed and the urine of the consumers is being used as feedstock for the VCD module.

Further work should be undertaken on TOC make-up. Total volatiles should be quantitated by running standards of the components of interest where possible. Non-strippable components should be determined by solvent extraction of the water samples after stripping.

7.0 SUMMARY

The first part of this study consisted of an additional

evaluation of the specific ingredients required to adequately enhance the taste of reclaimed water for long term space flight use. Previous investigations had shown that perhaps only a few inorganic species need be added to the inorganic free reclaimed water to provide the desired taste enhancement. The goal of this additional evaluation was to determine the minimum number and optimum quantities of chemical species required. Synthetic water samples were prepared by adding various cations and anions to quadruply distilled ultrapure water. The synthetic water samples were then evaluated by taste panels. The results of these tests indicate that two to four of the cations Ca^{++} , Mg^{++} , Na^+ , or K^+ in the concentration range 2-20 mg/L and either Cl^- or NO_3^- anion in stoichiometric proportion are sufficient for taste enhancement. Final selection of the proper mix of these cations should probably be left up to the actual spacecraft crew. The proper amounts of a concentrated stock solution of the desired species can be added to the portion of the reclaimed water destined for drinking on-board the spacecraft.

The second part of this study involved qualitative and semi-quantitative analysis of the volatile organic compounds in the product water of the Space Station Prototype Vacuum Compression Distillation (VCD) unit for the purpose of evaluating the process and the product water. Feedstock urine and water

samples were collected at various stages of processing. The volatiles were inert gas stripped, concentrated on Tenax GC adsorbent traps, and analyzed on a gas chromatograph-mass spectrometer-data system (GC-MS-DS) using high resolution capillary columns. The results of these analyses indicate that many components in the feedstock urine are not adequately removed by the distillation process but are generally greatly reduced by the post treatment charcoal filtration. Toxic compounds were present in both the condensate product and post treated final product water. Several unidentifiable components were present in the final product water which are most likely toxic compounds present in the feedstock. There is also evidence for contamination of both the condensate and final product in that components were present not found in the feedstock or in too great a concentration to have come solely from the feedstock. These results point up the need for additional evaluation of the VCD unit during future operational testing.

8.0 APPENDICES

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8.1 Appendix I: Tables

LIST OF TABLES

1. Spacecraft Water Quality-Palatability Specifications
2. Composition of Synthetic Water Samples
3. Taste Results-Standard Method
4. Taste Results (Hedonic Scale)
5. GC-MS Results for Urine + Pretreat Sample
6. GC-MS Results for Recycle Fluid
7. GC-MS Results for Untreated Condensate
8. GC-MS Results for VCD Laboratory Distilled Water
9. GC-MS Results for Final Product Water
10. Comparison of Individual Component Concentrations

TABLE 1
SPACECRAFT WATER QUALITY-PALABILITY SPECIFICATIONS

APPEARANCE

Color - 5 on cobalt scale

Total solids - less than 500 ppm

PALATABILITY

Dissolved gases

CO₂: 1-5 ppm

O₂: 1-5 ppm

Chemical Constituents - Inorganic

Ca ⁺⁺ :	20±5 ppm	Cl ⁻ :	30±10 ppm
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Mg ⁺⁺ , Na ⁺	10±2 ppm	SO ₄ ⁼ , NO ₃ ⁼ :	40±10 ppm
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K ⁺ :	2±1 ppm	HCO ₃ ⁼ , CO ₃ ⁼ :	10±5 ppm
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Chemical Constituents - Organic

Nonvolatile

Total Organic Carbon (TOC):	1 ppm
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Dissolved Organic Carbon (DOC):	1 ppm
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Particulate Organic Carbon (POC):	1 ppm
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Volatile

Total Headspace Extractable:	1 ppb
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Single Headspace Extractable:	To be determined
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TABLE 2
COMPOSITION OF SYNTHETIC WATER SAMPLES

<u>Sample No.</u>	<u>Ca⁺⁺</u>	<u>Mg⁺⁺</u>	<u>K⁺</u>	<u>Na⁺</u>	<u>Cl⁻</u>	<u>SO₄⁼</u>	<u>DO</u>
1	20	10	2	10	82	0.1	7.6
2	20	10	2	10	≤0.1	111	7.8
3	20	10	2	10	52	40	7.7
4	20	10	≤0.1	10	80	≤0.1	7.9
5	20	10	2	≤0.1	66	≤0.1	7.8
6	20	10	≤0.1	≤0.1	65	≤0.1	7.9
7	20	≤0.1	≤0.1	10	51	≤0.1	7.8
8	80	≤0.1	≤0.1	40	202	≤0.1	7.8
9	40	≤0.1	≤0.1	20	101	≤0.1	7.9
10	≤0.1	≤0.1	≤0.1	≤0.1	≤0.1	≤0.1	8.0

Note: All values in mg/L (ppm)

TABLE 3
TASTE RESULTS - STANDARD METHOD
(Paired with ultrapure water)

<u>Sample No.</u>	<u>Ratings</u>	<u>Ave. \pm S.D.</u>
1	6, 6, 6, 6, 6	6.0 \pm 0
2	3, 1, 2, 4, 7	3.4 \pm 2.6
3	5, 4, 5, 6, 7	5.4 \pm 1.3
4	6, 7, 5, 6, 7	6.2 \pm 0.9
5	4, 10, 5, 2, 7	5.6 \pm 3.4
6	4, 2, 5, 4, 7	4.4 \pm 2.2
7	4, 6, 6, 6, 6	5.6 \pm 0.9
8	3, 5, 6, 6, 4	4.8 \pm 1.2
9	4, 5, 6, 5, 8	5.6 \pm 1.7
10	Assigned a value of 5	

TABLE 4
TASTE RESULTS
(Hedonic Scale)

A. Standard Method

<u>Sample No.</u>	<u>Ratings</u>	<u>Ave. \pm S.D.</u>
1	4, 6, 4, 7, 5	5.2 \pm 1.3
2	4, 4, 0, 6, 2	3.2 \pm 2.6
3	3, 6, 3, 6, 4	4.4 \pm 1.3
4	4, 5, 5, 7, 5	5.2 \pm 1.3
5	5, 5, 5, 7, 5	5.4 \pm 0.9
6	4, 5, 6, 7, 5	5.4 \pm 1.3
7	5, 4, 6, 6, 5	5.2 \pm 0.9
8	7, 6, 4, 7, 6	6.0 \pm 1.3
9	6, 5, 4, 6, 5	5.2 \pm 0.9
10	5, 4, 4, 5, 5	4.6 \pm 0.4

B. After Swallowing

<u>Sample No.</u>	<u>Ratings</u>	<u>Ave. \pm S.D.</u>
1	5, 6, 5, 7, 5	5.6 \pm 0.9
2	4, 3, 0, 7, 2	3.2 \pm 3.0
3	5, 7, 3, 5, 3	4.6 \pm 1.7
4	5, 5, 5, 7, 5	5.4 \pm 1.3
5	5, 4, 5, 8, 4	5.2 \pm 1.7
6	5, 5, 6, 7, 6	5.8 \pm 0.9
7	5, 3, 6, 6, 5	5.0 \pm 1.3
8	6, 6, 4, 7, 7	6.0 \pm 1.3
9	6, 5, 4, 6, 5	5.2 \pm 0.9
10	5, 4, 4, 5, 5	4.6 \pm 0.4

TABLE 5

GC-MS RESULTS FOR URINE + PRETREAT SAMPLE

Scan	Identification	Formula	Wt. %	Estimated Amount (x 10 ⁻⁹ g)
69	acetone	CH ₃ COCH ₃	.068	170
73	Methyl iodide	CH ₃ I	2.76	7020
88	Ethyl iodide	C ₂ H ₅ I	.086	219
104	3-Methyl-2-butanone	C ₅ H ₁₀ O		
	Benzene	C ₆ H ₆	.069	176
	Carbon tetrachloride	CCl ₄		
133	1,4-dioxane	C ₄ H ₈ O ₂	4.34	11,000
159	dimethyl disulfide + 1,3 dioxane	CH ₃ S-S-CH ₃	1.21	3,080
163- 180	C ₅ alcohols	C ₅ H ₁₂ O	6.54	16,600
185	toluene	C ₇ H ₈	0.285	724
210	branched paraffin	C ₉ H ₂₀	0.087	221
258	3-ethyl-2-methyl pentane	C ₉ H ₂₀	0.043	108
275	4-ethyl heptane	C ₉ H ₂₀	0.052	132
302	hexamethyl cyclo trisiloxane	C ₆ H ₁₈ O ₃ Si ₃	0.702	1,780
354	butyl iodide	C ₄ H ₉ I	4.34	11,000
506	2-furyl methyl ketone	C ₆ H ₆ O ₂	0.272	690
545	2,3,4 trithiapentane	C ₃ H ₆ S ₃	0.639	1,620
566	olefin	C _n H _{2n}	0.043	110
582	benzaldehyde	C ₇ H ₆ O	0.031	78
623	heptanol	C ₇ H ₁₆ O	0.484	1,230
662	6-methyl-5-nonen-4-one	C ₁₀ H ₁₈ O	0.186	472
673	isopropyl toluene (p-cymene)	C ₁₀ H ₁₄	0.391	991

TABLE 6

GC-MS RESULTS FOR RECYCLE FLUID

Scan	Identification	Formula	Wt. %	Estimated _g Amount (x10 ⁻⁹ g)
83	1,4 Dioxane	C ₄ H ₈ O ₂	11.27	12,000
139	1,3 Dioxane	C ₄ H ₈ O ₂	3.76	4,000
210- 312	C ₅ alcohols	C ₅ H ₁₂ O	16.90	18,000
351	hexamethyl cyclotrisiloxane	C ₆ H ₁₈ O ₃ Si ₃	9.39	10,000
464	not identified		0.464	490
497	2-furyl methyl ketone	C ₆ H ₆ O ₂	6.30	6,710
559	2,3,4 trithiapentane	C ₃ H ₆ S ₃	2.56	2,730
587	2-methyl-3-ethyl heptane	C ₁₀ H ₂₂	0.072	77
665	6-methyl-5-nonen-4-one	C ₁₀ H ₁₈ O	0.175	186
678	isopropyl toluene (p-cymene)	C ₁₀ H ₁₄	2.42	2,580
697	octamethyl cyclotetrasiloxane	C ₈ H ₂₄ O ₄ Si ₄	14.08	15,000
778	dienyl ketone	C ₁₀ H ₁₆ O	0.142	152
814	cyclic alcohol	C ₁₀ H ₂₀ O	0.552	588
846	dimethyl styrene	C ₁₀ H ₁₂	3.43	3,650
928	methoxy phenol	C ₇ H ₈ O ₂	0.093	99
957	1-acetyl-2,3-dimethyl cyclobutene	C ₈ H ₁₂ O	0.088	94
976	o-cresol	C ₇ H ₈ O	3.91	4,160
1043	not identified		0.064	68
1058	p-cresol	C ₇ H ₈ O	0.208	222
1109	trimethyl-1-indanone	C ₁₂ H ₁₄ O	0.139	148
1123	decamethyl cyclopentasiloxane	C ₁₀ H ₃₀ O ₅ Si ₅	19.82	21,000
1136	trimethyl tetrahydronaphthalene	C ₁₃ H ₁₈	3.47	3,690

TABLE 6
GC-MS RESULTS FOR RECYCLE FLUID

-Continued-

<u>Scan</u>	<u>Identification</u>	<u>Formula</u>	<u>Wt. %</u>	<u>Estimated Amount (x10⁻⁹ g)</u>
1215	alpha-ionone	C ₁₃ H ₂₀ O	0.423	450
1243	a siloxane		0.068	72
1264	not identified		0.133	142
1277	not identified		0.118	126
1293	trimethyl dihydronapthalene	C ₁₃ H ₁₆	0.138	147
1318	decamethyl cyclohexasiloxane	C ₁₀ H ₃₄ O ₆ Si ₆	9.19	<u>9,790</u>
			TOTAL	106,481

Estimated Total Volatile Organic Content 7100 µg/L

TABLE 7

GC-MS RESULTS FOR UNTREATED CONDENSATE

Scan	Identification	Formula	Wt. %	Estimated Amount ($\times 10^{-9}$ g)
103	Acetone	C_3H_6O	0.089	59
108	Methylene chloride	CH_2Cl_2	0.033	22
114	Ethyl alcohol	C_2H_6O	0.17	133
120	Ethyl sec-butyl ether	$C_6H_{14}O$	0.12	80
177	Propyl alcohol	C_3H_8O	2.62	1,720
213	1,4 Dioxane	$C_4H_8O_2$	12.97	8,510
238	C_5 alcohol	$C_5H_{12}O$	0.14	93
248	C_5 alcohol	$C_5H_{12}O$	0.18	116
253	Dimethyl disulfide	$C_2H_6S_2$	0.42	274
279	C_5 alcohol	$C_5H_{12}O$	53.36	35,000
375	Branched paraffin	C_9H_{20}	0.55	359
441	4-ethyl heptane	C_9H_{20}	0.093	61
484	hexamethyl cyclotrisiloxane	$C_6H_{18}O_3Si_3$	0.29	192
725	not identified		0.34	221
807	isopropyl toluene (p-cymene)	$C_{10}H_{14}$	0.19	125
818	octamethyl cyclo tetrasiloxane	$C_8H_{24}O_4Si_4$	0.15	97
864	2-ethyl-1-hexanol	$C_8H_{18}O$	0.39	255
881	4-t butyl cyclohexanone	$C_{10}H_{18}O$	0.043	28
908	dimethyl styrene	$C_{10}H_{12}$	0.15	100
1175	menthol	$C_{10}H_{20}O$	26.98	17,700
1478	dodecamethyl cyclo hexasiloxane	$C_{12}H_{36}O_6Si_6$	0.62	408
1700	undecamethyl cyclo hexasiloxane	$C_{11}H_{34}O_6Si_6$	0.09	62
			TOTAL	65,595

Estimated Total Volatile Organic Content 1310 μ g/L

TABLE 8
GC-MS RESULTS FOR VCD LABORATORY DISTILLED WATER

<u>Scan</u>	<u>Identification</u>	<u>Formula</u>	<u>Wt. %</u>	<u>Estimated^d Amount (x10⁻⁹ g)</u>
108 -113	Acetone	C ₃ H ₇ O	34.7	2,880
151	Benzene	C ₆ H ₆	65.3	<u>5,420</u>
			TOTAL	8,300

Estimated Total Volatile Organic Content 166µg/l

TABLE 9
GC-MS RESULTS FOR FINAL PRODUCT WATER

<u>Scan</u>	<u>Identification</u>	<u>Formula</u>	<u>Wt. %</u>	<u>Estimated Amount (x10⁻⁹ g)</u>
96	acetone	C ₃ H ₇ O	7.0	4,500
97	methylene chloride	CH ₂ Cl ₂	7.0	4,500
106	ethyl alcohol	CH ₄ O	62.3	39,900
138	benzene	C ₆ H ₆	23.5	15,100
588	2-methyl-3-ethyl heptane	C ₁₁ H ₂₂	0.078	50
636	not identified		0.020	13
695	paraffin	C _n H _{2n+2}	0.043	28
724	not identified		0.021	14
892	not identified		0.010	7
939	not identified		0.020	13
1053	not identified		0.009	6
1065	not identified		0.010	7
1148	not identified		0.008	5
1404	not identified		0.010	7
1427	undecamethyl cyclohexasiloxane		0.037	<u>24</u>
			TOTAL	64,236

Estimated Total Volatile Organic Content 1280 µg/L

TABLE 10

COMPARISON OF INDIVIDUAL COMPONENT CONCENTRATIONS

<u>Compound</u>	<u>Estimated Concentration (ug/L as tetradecane)</u>			
	<u>Urine + Pretreat</u>	<u>Recycle Fluid</u>	<u>Untreated</u>	<u>Product</u>
Acetone	3.4	ND	1.2	90
Methylene chloride	ND	ND	0.4	90
Methyl iodide	140	ND	ND	ND
Ethyl iodide	4.4	ND	ND	ND
Ethyl alcohol	ND	ND	2.3	800
3-Methyl-2-butanone	1.2	ND	ND	ND
Benzene	1.2	ND	ND	302
Carbon Tetrachloride	1.2	ND	ND	ND
Ethyl sec-butyl ether	ND	ND	1.6	ND
Propyl alcohol	ND	ND	34	ND
1,4 dioxane	220	800	170	ND
1,3 dioxane	62	270	ND	ND
dimethyl disulfide	62	ND	5.5	ND
toluene	14	ND	ND	ND
C ₅ alcohols	330	1200	705	ND
branched C ₉ paraffin	4.4	ND	7.2	ND
3-ethyl-2-methyl pentane	2.1	ND	ND	ND
4-ethyl heptane	2.6	ND	1.2	ND
hexamethyl cyclo trisiloxane	36	670	3.8	ND
butyl iodide	220	ND	ND	ND
2-furyl methyl ketone	14	450	ND	ND
2,3,4 trithiapentane	32	180	ND	ND
olefin	2.2	ND	ND	ND
benzaldehyde	1.6	ND	ND	ND
2-methyl-3-ethyl heptane	ND	5.1	ND	1.0
heptanol	24.5	ND	ND	ND
6-methyl-5-nonen-4-one	9.4	12	ND	ND
isopropyl toluene	20	170	2.5	ND
octamethyl cyclotetrasiloxane	1350	1000	1.9	ND
dienyl ketone	14	10	ND	ND
C ₁₀ cyclic alcohol	14	39	ND	ND
2-ethyl-1-hexanol	ND	ND	5.1	ND
4-tertbutyl cyclohexanone	ND	ND	0.5	ND
dimethyl styrene	125	240	2.0	ND
methoxy phenol	ND	6.6	ND	ND
1-acetyl-2,3-dimethyl cyclobutene	ND	6.3	ND	ND
O-cresol	ND	780	ND	ND
p-cresol	ND	15	ND	ND

TABLE 10
COMPARISON OF INDIVIDUAL COMPONENT CONCENTRATIONS
-Continued-

<u>Compound</u>	<u>Estimated Concentration (ug/L as tetradecane)</u>			
	<u>Urine + Pretreat</u>	<u>Recycle Fluid</u>	<u>Untreated</u>	<u>Product</u>
trimethyl-1-indonone	ND	9.9	ND	ND
decamethyl cyclopentasiloxane	1360	1400	ND	ND
trimethyl tetrahydronaphthalene	ND	250	ND	ND
alpha ionone	17	30	ND	ND
menthol	ND	ND	355	ND
C ₁₆ diene	7.0	ND	ND	ND
trimentyl dihydronaphthalene	4.2	9.8	ND	ND
dodecamethyl cyclohexasiloxane	820	650	8.2	ND
undecamethyl cyclohexasiloxane	150	ND	1.2	0.5
Estimated Total Volatile Organics (ug/L or ppb)	5070	7100	1310	1280

8.2 Appendix II: Figures

LIST OF FIGURES

1. Taste of Synthetic Water Samples
2. Schematic Diagram of SSP VCD Module
3. Tenax Trap and Injector Port
4. Aqueous Volatile Sampler
5. Sample Analysis and Transfer Flow System
6. Urine + Pretreat RGC
7. Recycle Fluid RGC
8. Untreated Product RGC
9. Distilled Water Blank RGC
10. Product Water RGC
11. Urine + Pretreat Expanded RGC
12. Recycle Fluid Expanded RGC
13. Untreated Product Expanded RGC
14. Distilled Water Expanded RGC
15. Product Water Expanded RGC

Figure 1
TASTE OF SYNTHETIC WATER SAMPLES

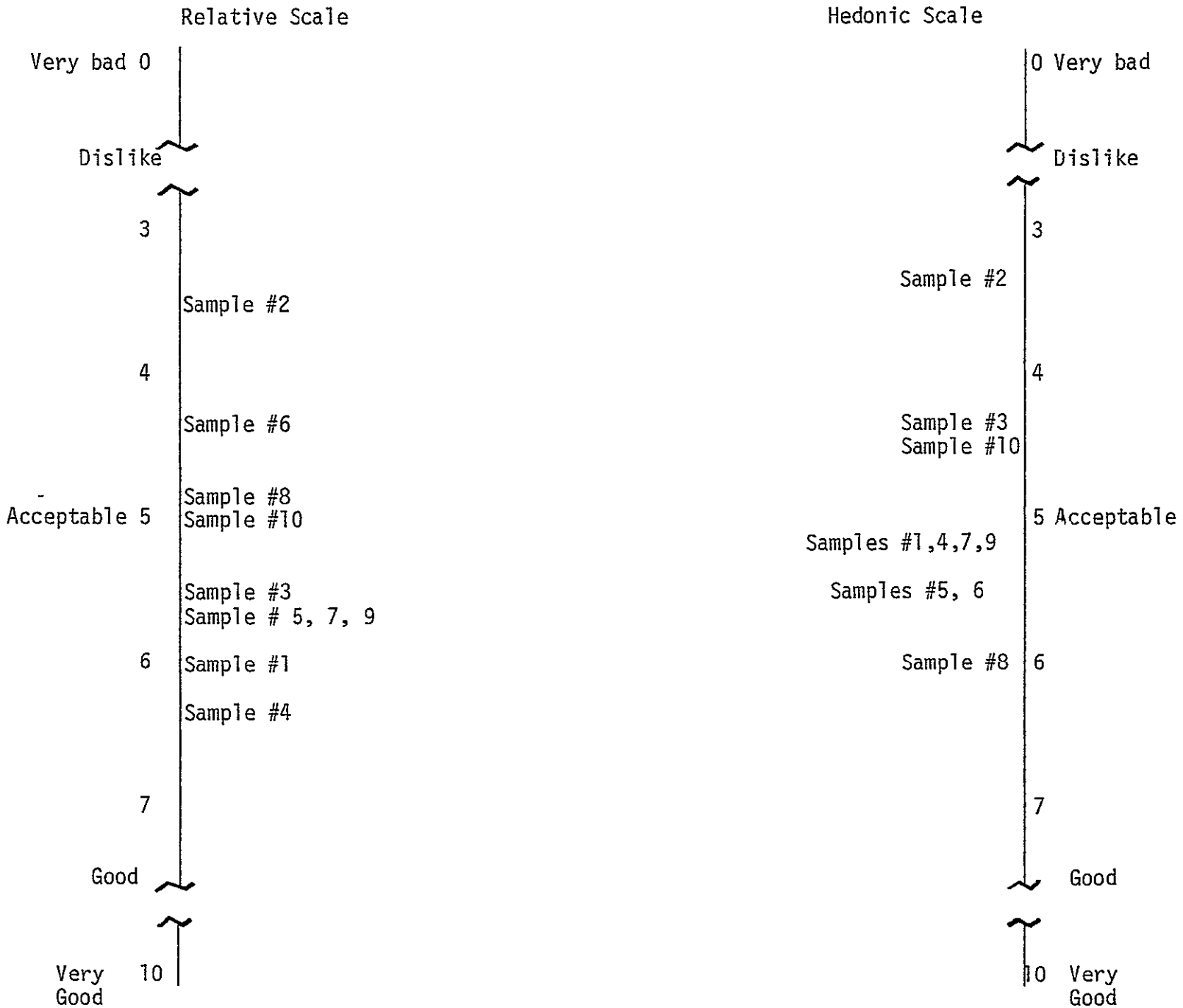




Figure 2 SCHEMATIC DIAGRAM OF SSP VCD MODULE

Figure 3

MODIFIED INJECTOR PORT FOR SAMPLER DESORPTION

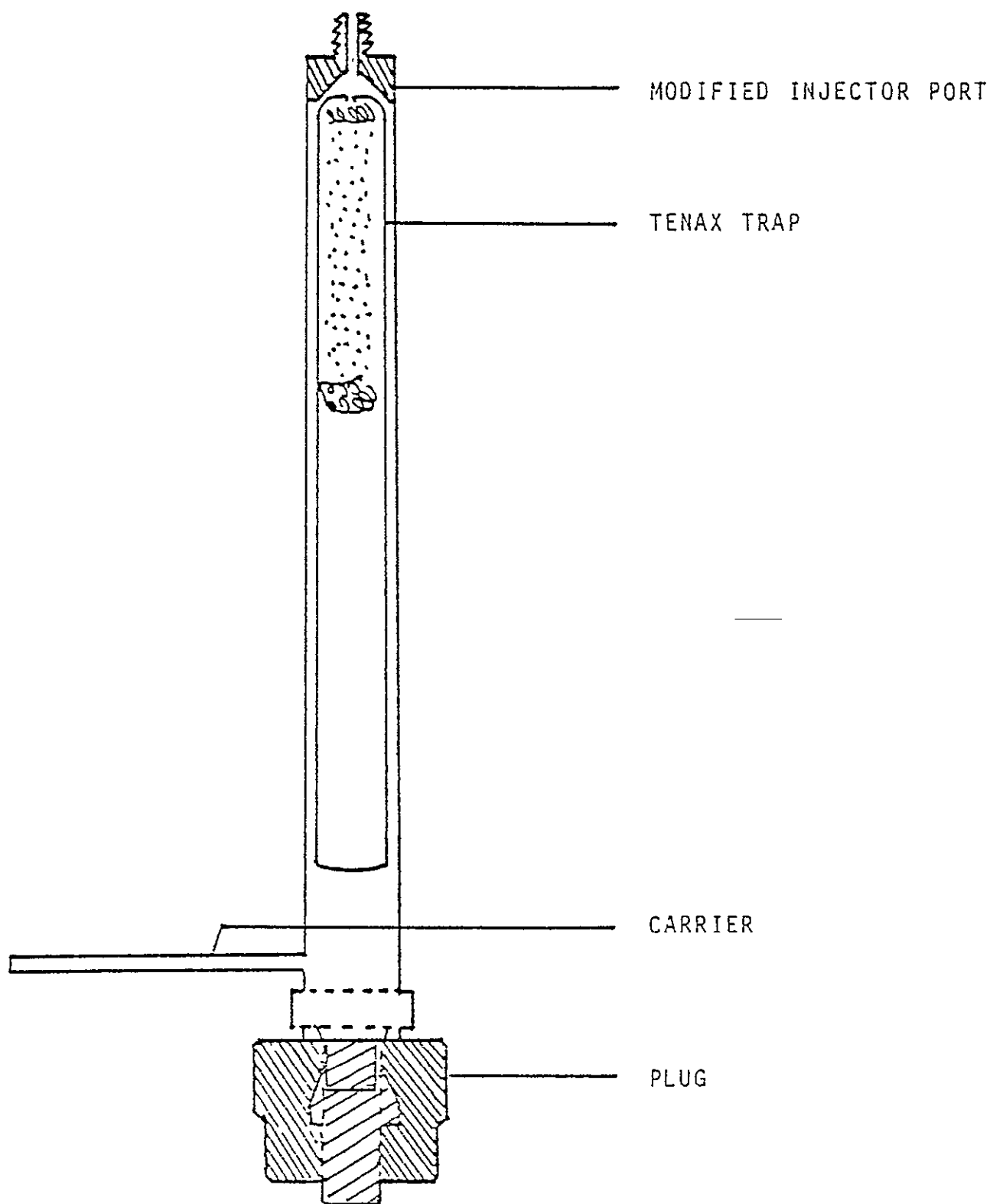
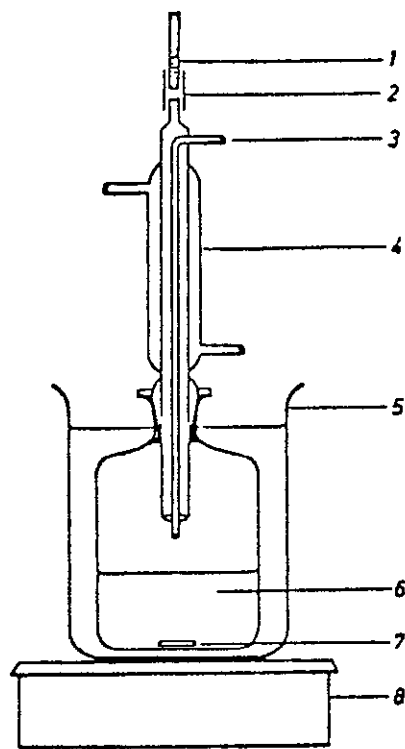


Figure 4-Aqueous Volatile Sampler



● Aqueous Volatile Sampler

1 Trap-insert with Tenax-GC, 2 Teflon sleeve, 3 Helium inlet,
4 Condenser, 5 Boiling water bath, 6 Sample, 7 Magnetic stirring
bar, 8 Hot-plate-stirrer

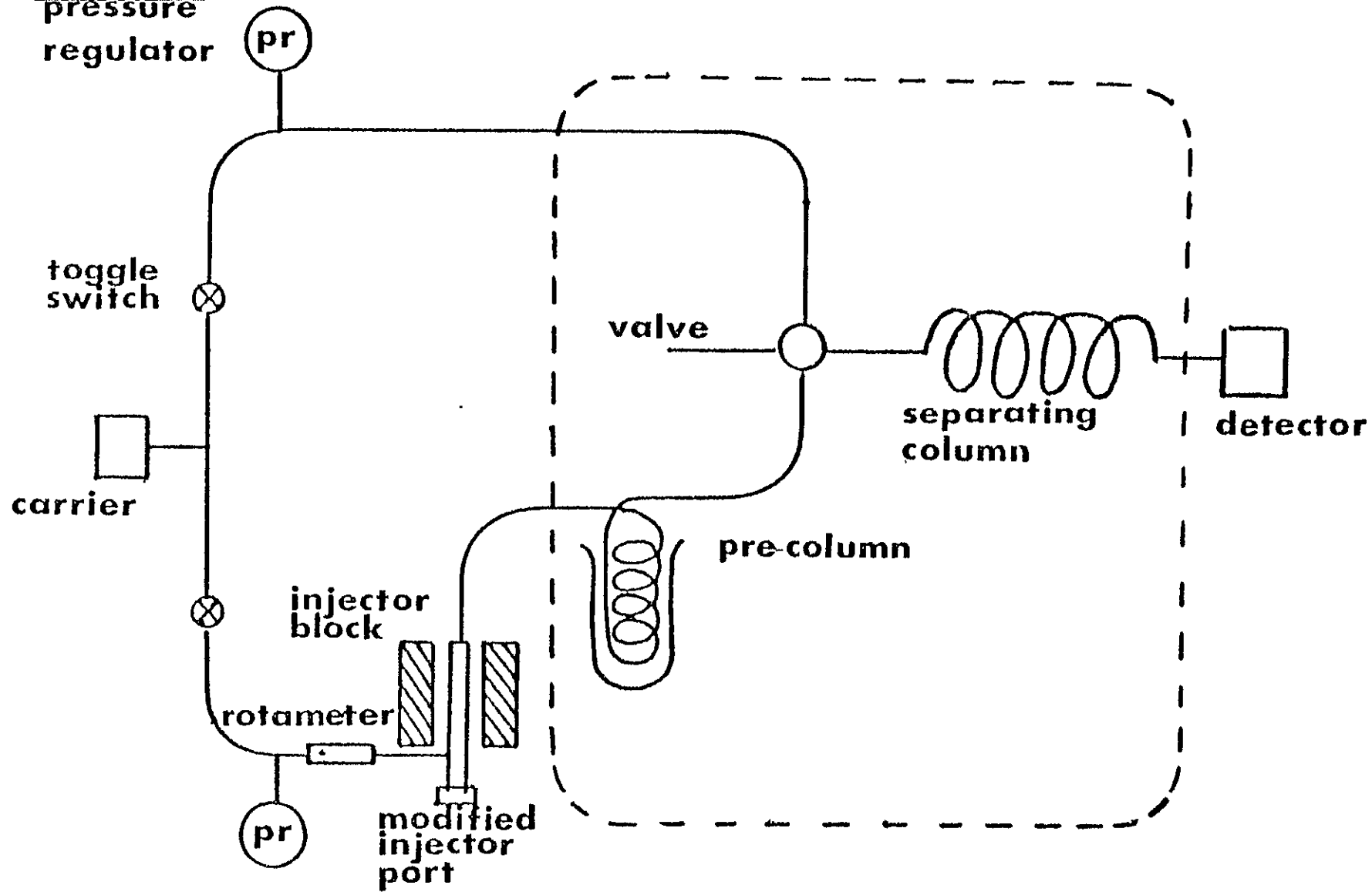


Figure 5

SAMPLE TRANSFER AND ANALYSIS FLOW SYSTEM

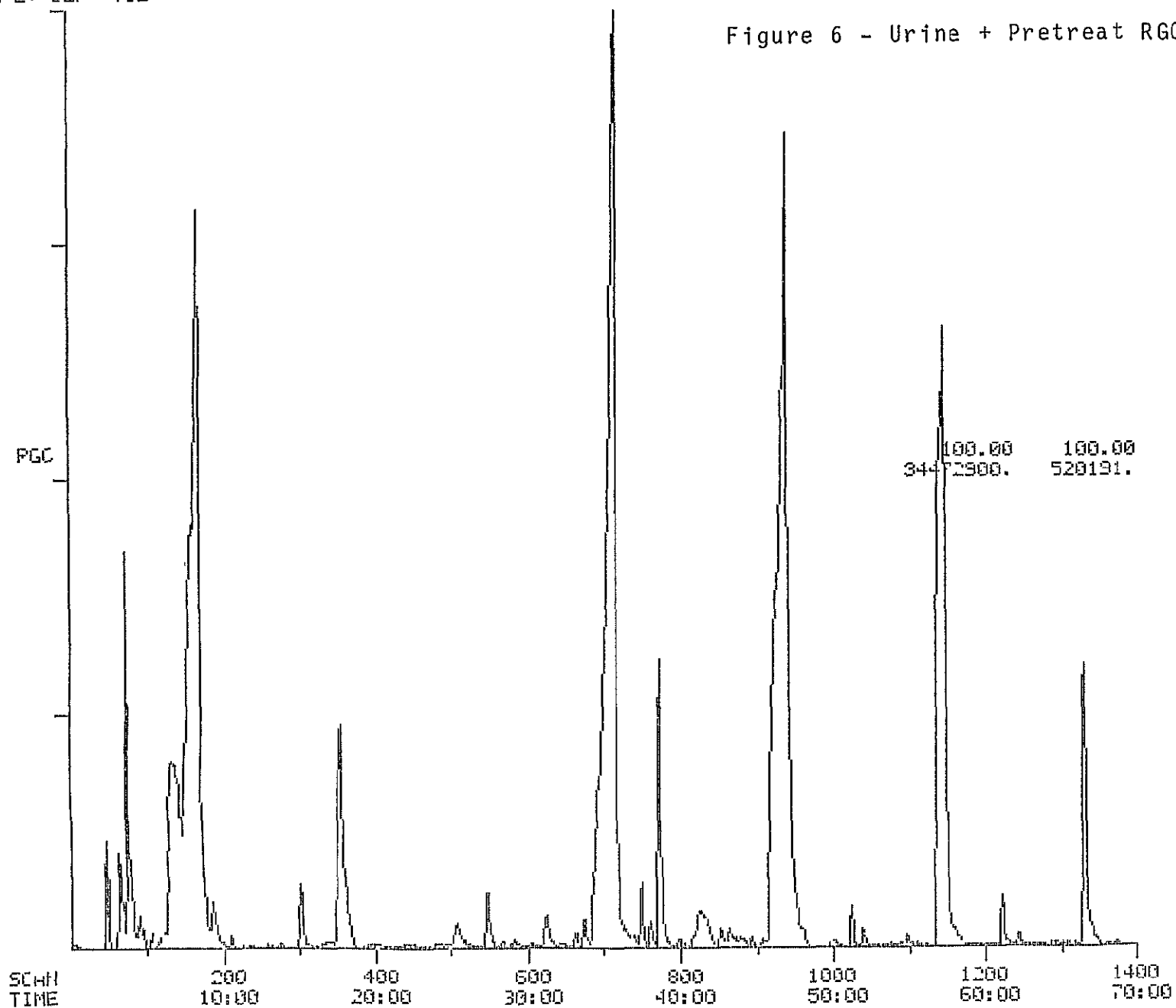
RECONST. GAS CHROMATOGRAM
DATE: 01.11.76 TIME: 916
SAMPLE: AIP

SAMPLE RUN: 11913 SCHMS 1 TO 1400
CALIB. RUN: C011077PP18

M E. DEF. TOL

CP.H. .CPGC.HPEH CP.H. .CPGC.HEIGHT

Figure 6 - Urine + Pretreat RGC



RECONST. GAS CHROMATOGRAM
DATE: 01/11/76 TIME: 1135
SAMPLE: TENH:12 RECYCLE

SAMPLE RUN: 11925 SCHS 1 TO 1400
CALIB. RUN: C011077PP18

M/E, DEF, TOL

%P.H. %P.GC .H.PEH %P.H. %P.GC .HEIGHT

Figure 7 - Recycle Fluid RGC

RGC

Retention Time (min)	Height
100.00	1846000.
130.00	291327.

SCAN
TIME

200 10:00 400 20:00 600 30:00 800 40:00 1000 50:00 1200 60:00 1400 70:00

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RECONST. GAS CHROMATOGRAM
 DATE: 01-11-76 TIME: 1453
 SAMPLE: TENH/13 UNTREATED

SAMPLE RUN: 11935 SCANS 1 TO 1750
 CALIB. RUN: C011077PP18

P.P.H. .NPGC.HPEH P.P.H. .NPGC.HEIGHT

Figure 8 - Untreated Product RGC

M/E. DEF. TOL

RGC

100.00 100.00
 9814440. 258303.

SCAN TIME 200 400 600 800 1000 1200 1400 1600
 10:00 20:00 30:00 40:00 50:00 60:00 70:00 80:00

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 OF POOR QUALITY

RECONST. GAS CHROMATOGRAPH
DATE: 01-13-77 TIME: 13
SAMPLE: BLANK TENAX 35

SAMPLE RUN: 11945 SCHMS 1 TO 1800
CHLIB. RUN: C011077PP18

2P.H., 2PGC, AREA/2P.H., 2PGC, HEIGHT

Figure 9 - Distilled Water Blank RGC



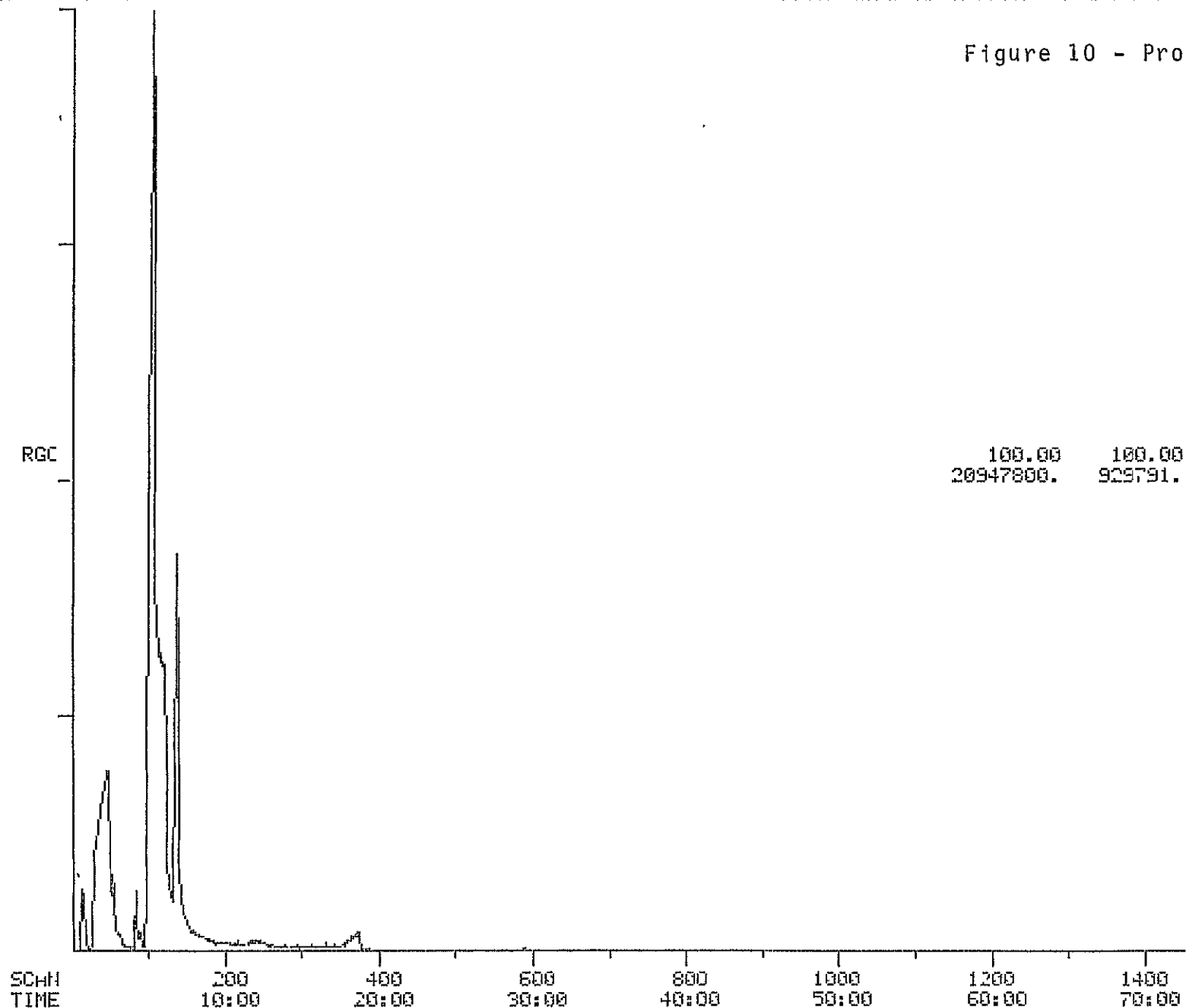
RECONST. GAS CHROMATOGRAM
DATE: 01 11-76 TIME: 2042
SAMPLE: HCD PRODUCT

SAMPLE RUN: 1195\$ SCHMS 1 TO 1450
CALIB. RUN: 0011077PP18

ZR.H. .ZRG. .HFEH .SP.A. .ZPGC .HEIGHT

M.E. DEF. TOL

Figure 10 - Product Water RGC

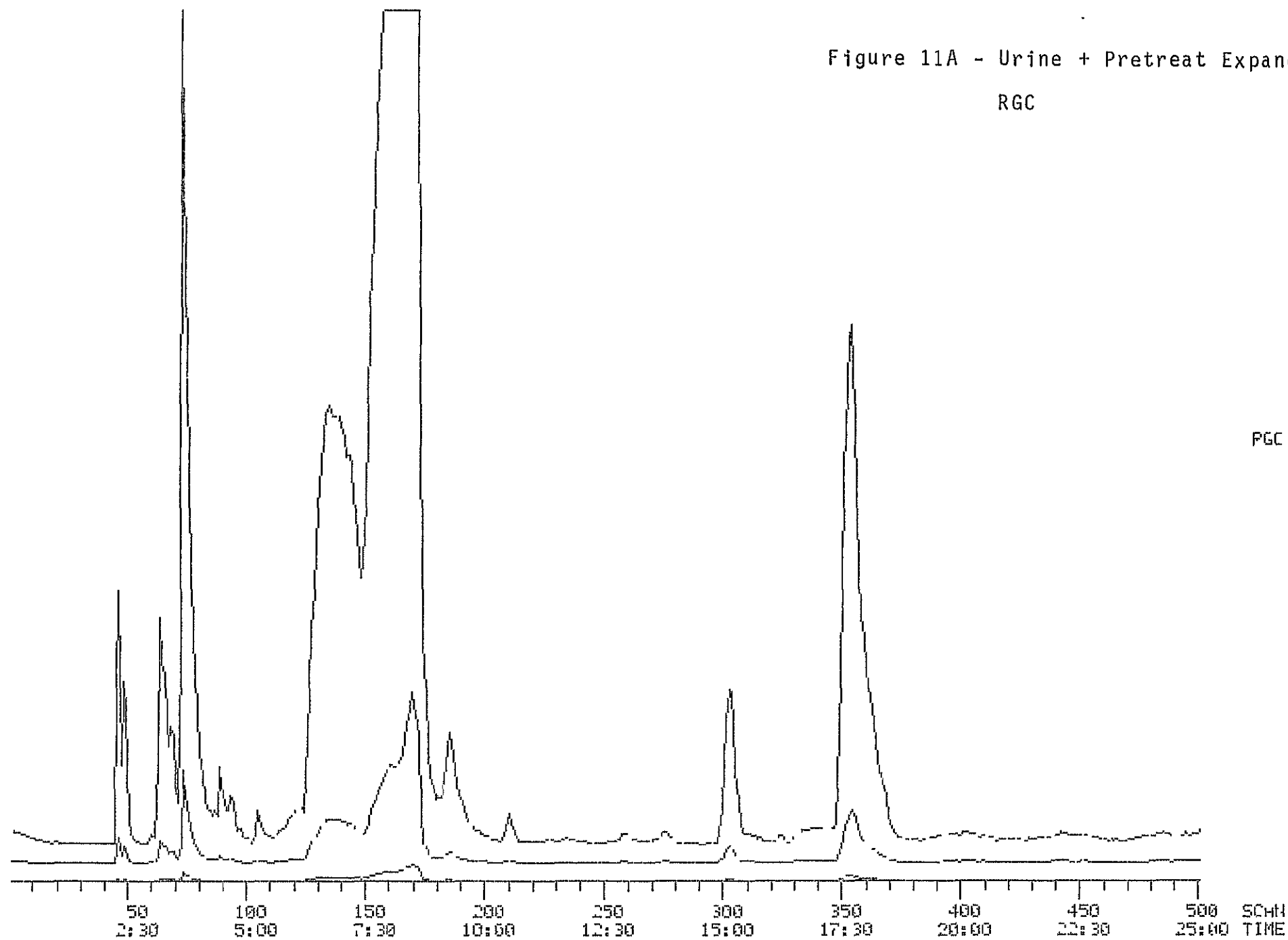


RGC
DATE: 01/11/76 TIME: 915
SAMPLE: AIP

SAMPLE RUN: 11915
CHLIB. RUN: C011077PP18

SCANS 1 TO 500

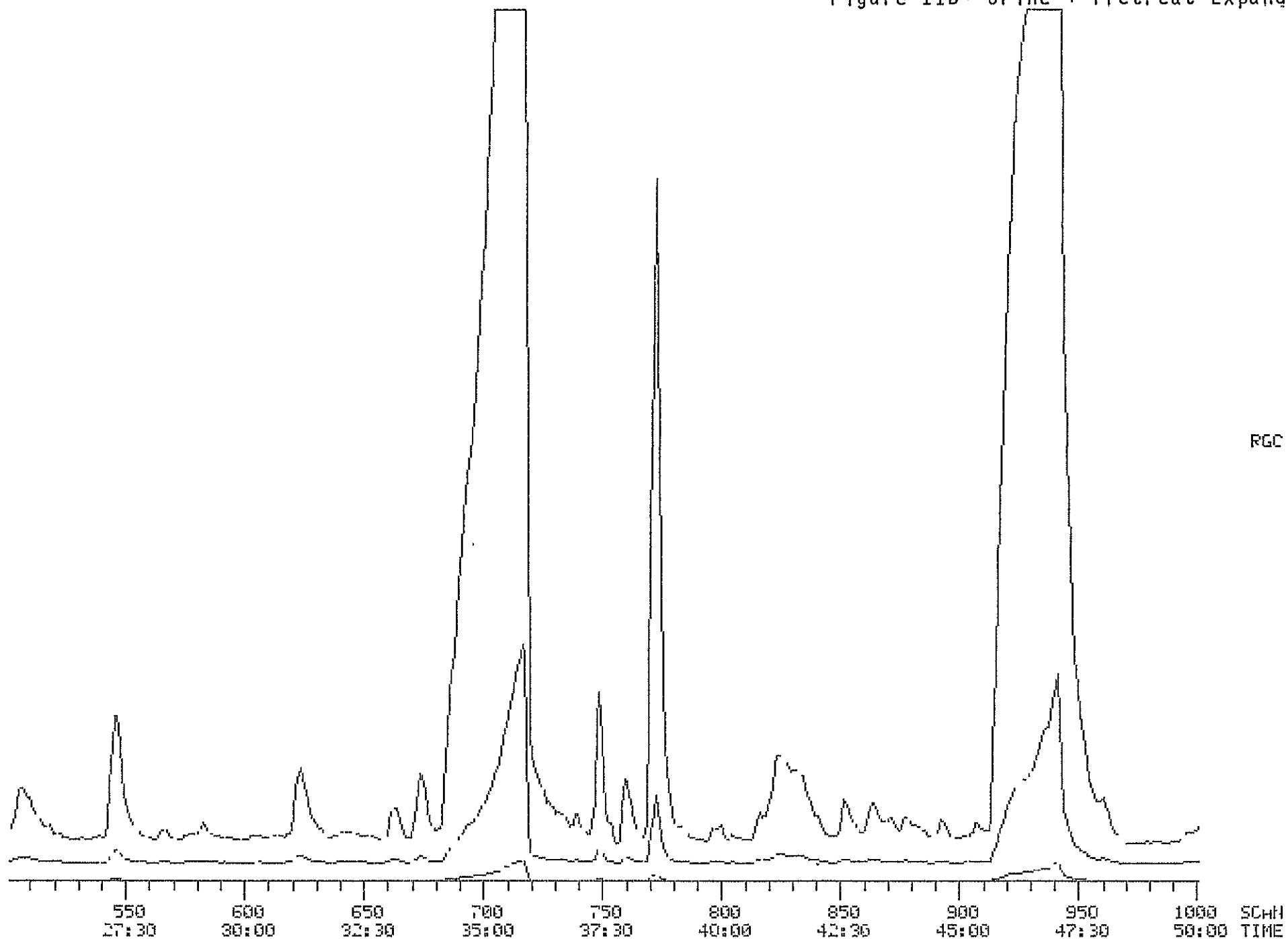
Figure 11A - Urine + Pretreat Expanded
RGC



RGC
DATE: 01/11/76 TIME: 916
SAMPLE: AIP

SAMPLE RUN: 11915 SCANS 501 TO 1000
CALIB. RUN: C011077PP18

Figure 11B—Urine + Pretreat Expanded RGC

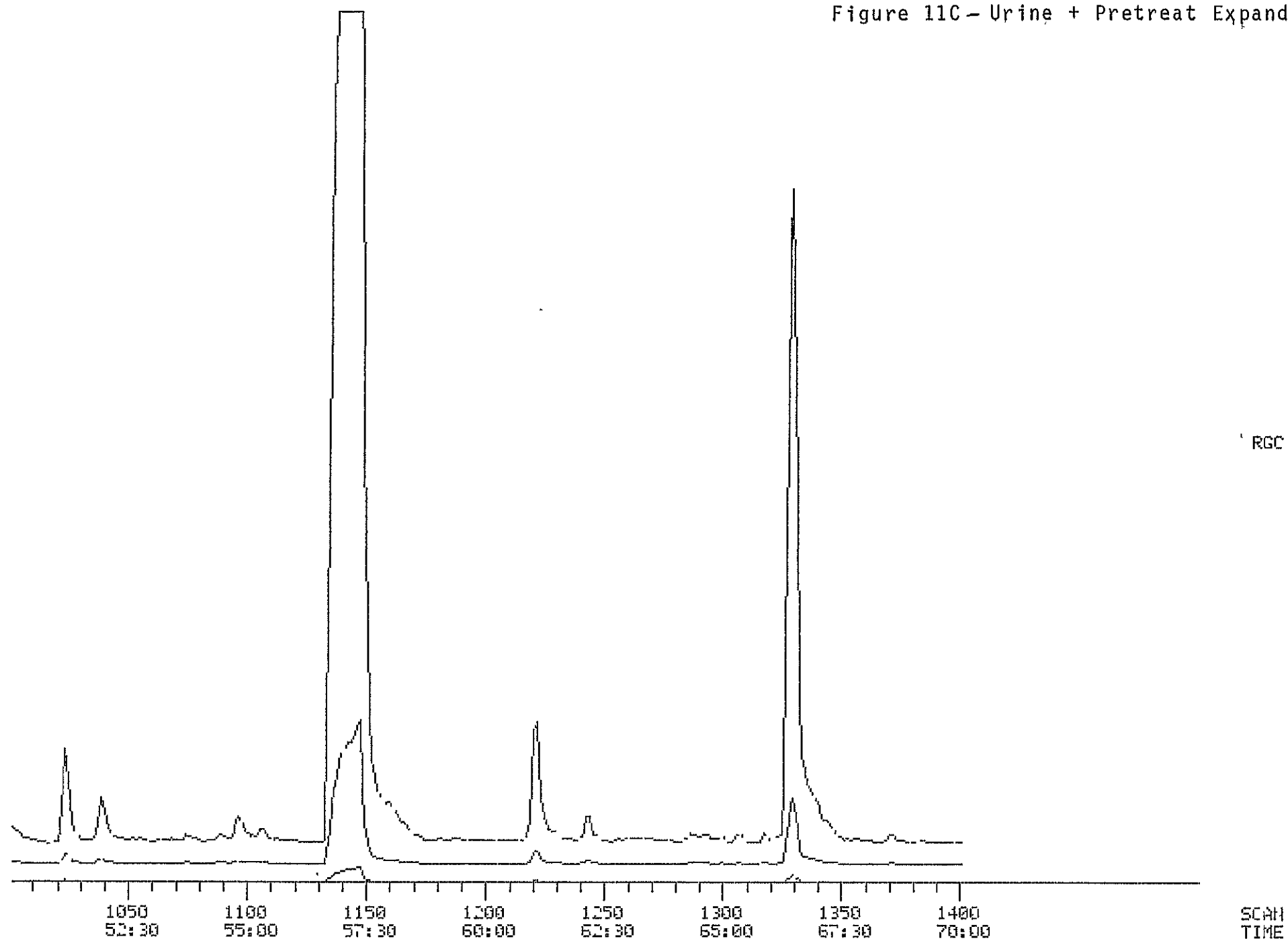


PGC
DATE: 01/11/76 TIME: 916
SAMPLE: H1P

SAMPLE RUN: 11915
CALIB. RUN: C011077PP18

SCANS 1001 TO 1400

Figure 11C - Urine + Pretreat Expanded RGC

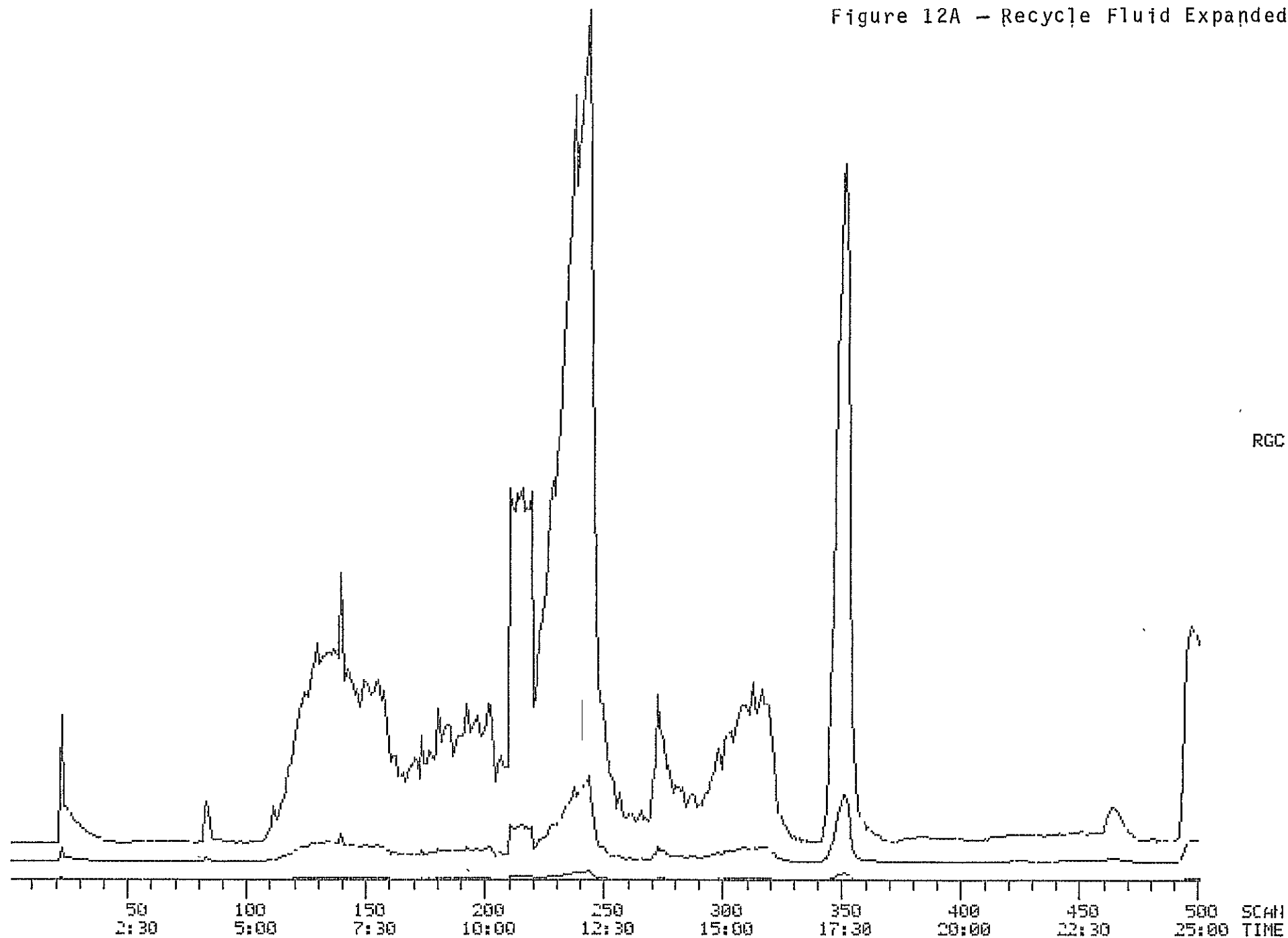


RGC
DATE: 01 11 76 TIME: 1135
SAMPLE: TENAN12 RECYCLE

SAMPLE RUN: 11925
CALIB. RUN: C011077PP18

SCANS 1 TO 500

Figure 12A - Recycle Fluid Expanded RGC

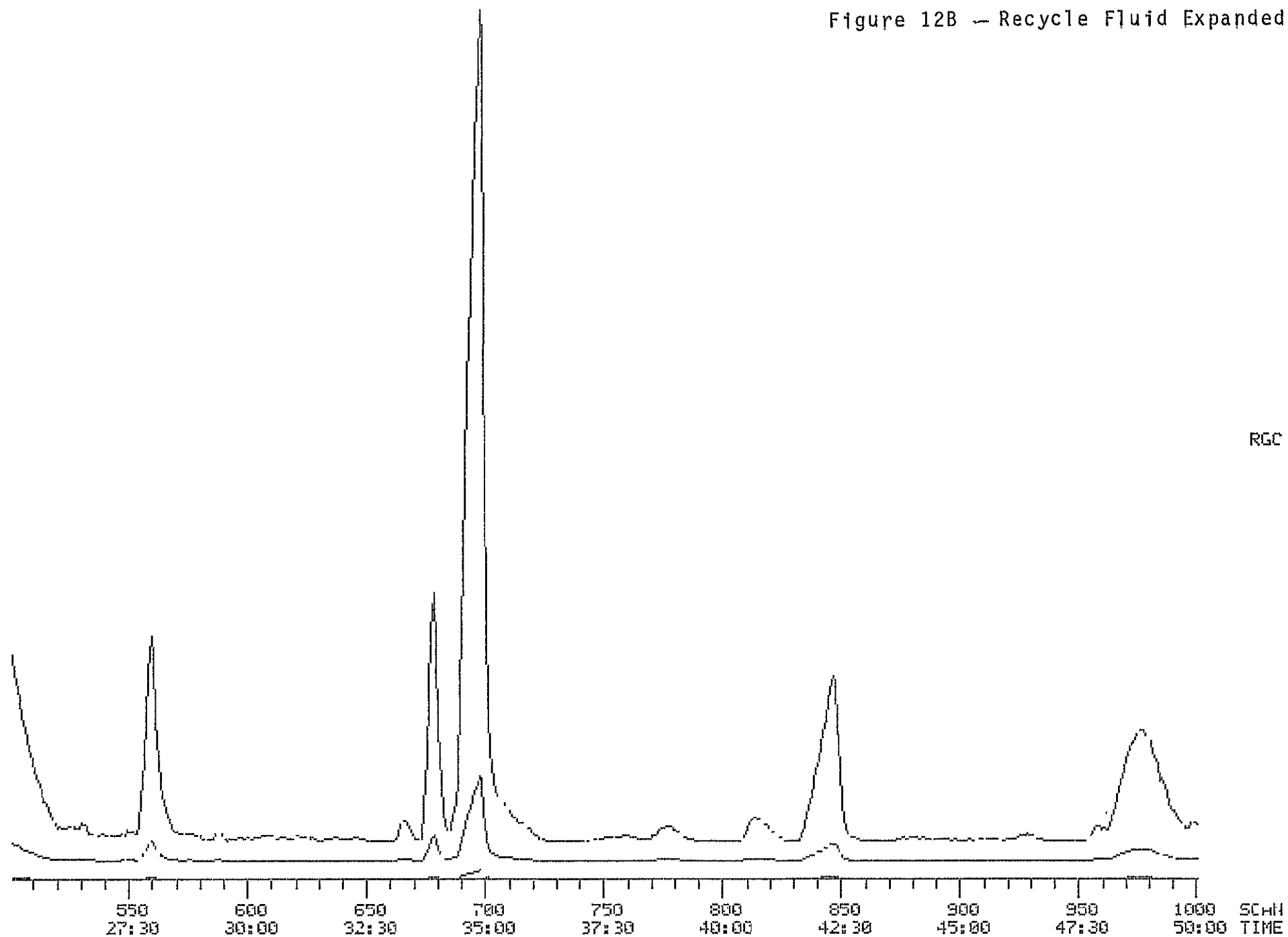


RGC
DATE: 01/11/76 TIME: 1135
SAMPLE: TENH:12 RECYCLE

SAMPLE RUN: 11925
CHLIB, PUH: C011077PP18

SCANS 501 TO 1000

Figure 12B — Recycle Fluid Expanded RGC

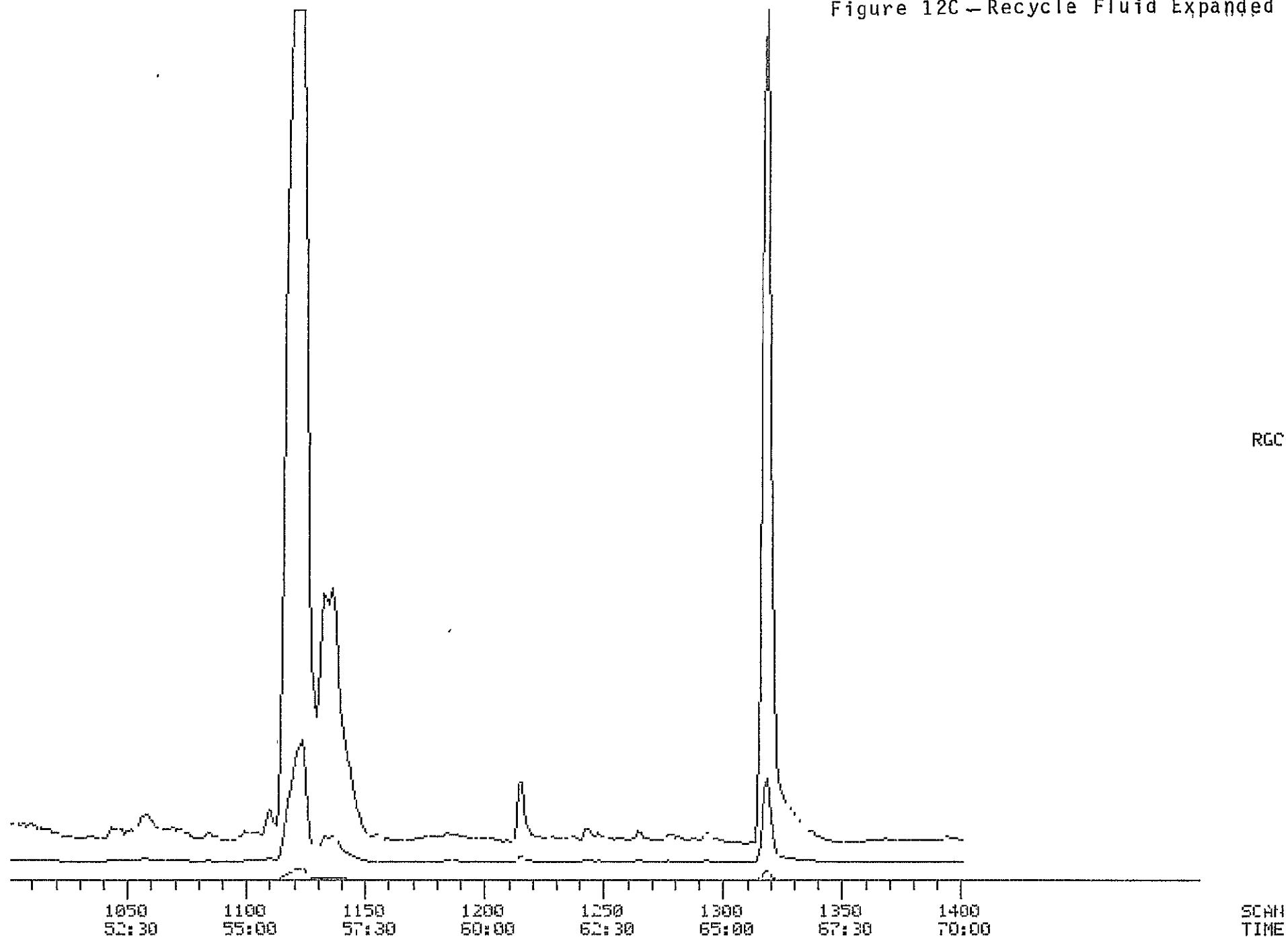


PGC
DATE: 01 11-76 TIME: 1135
SAMPLE: TENH:12 RECYCLE

SAMPLE RUN: 11925
CALIB. RUN: C011077FP18

SCANS 1001 TO 1400

Figure 12C - Recycle Fluid Expanded RGC

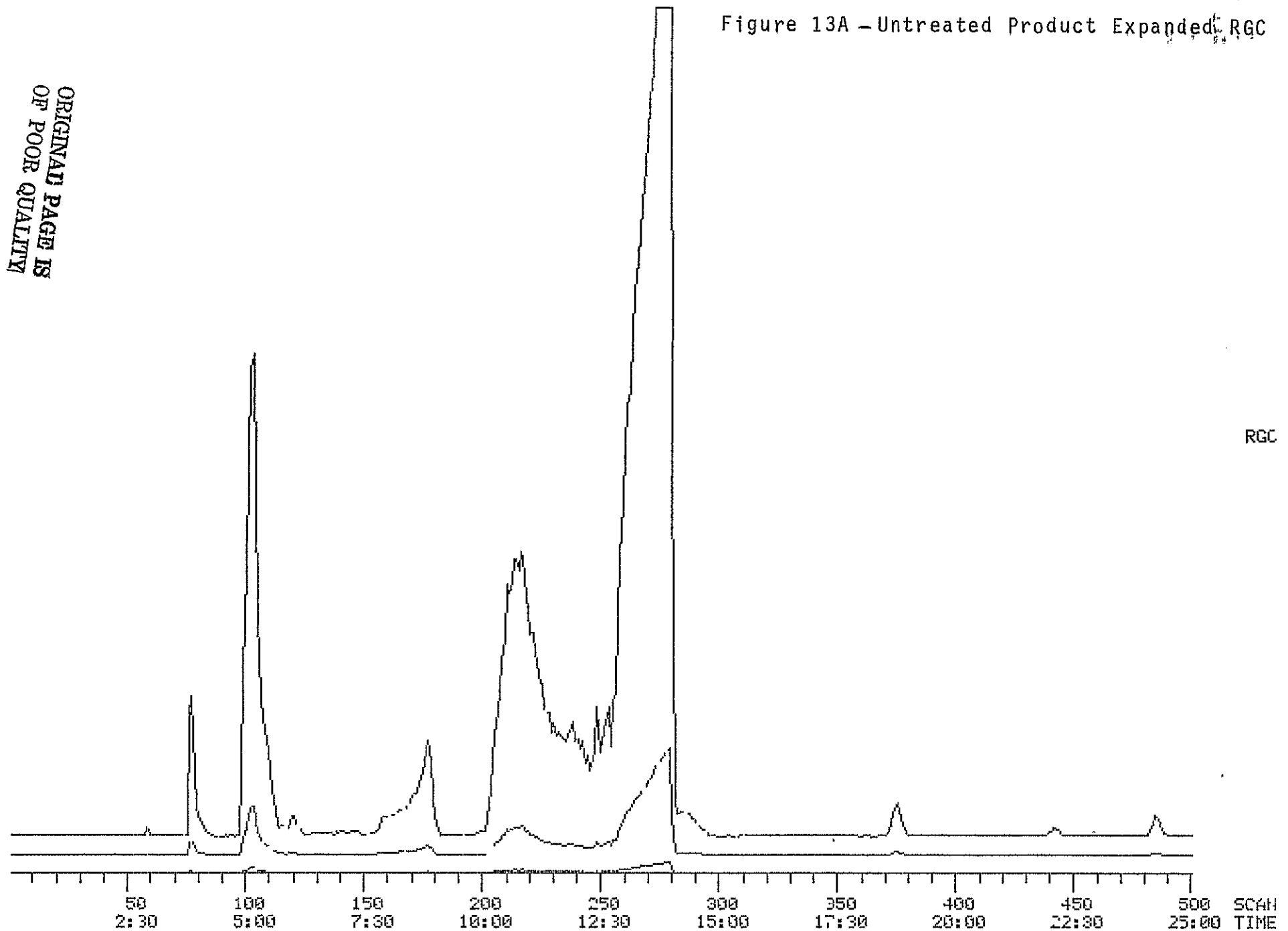


RGC
DATE: 01-11-76 TIME: 1453
SAMPLE: TENH-13 UNTREATED

SAMPLE RUN: 11935
CHLIB. RUN: C011077PP18

SCHMS 1 TO 500

Figure 13A - Untreated Product Expanded RGC



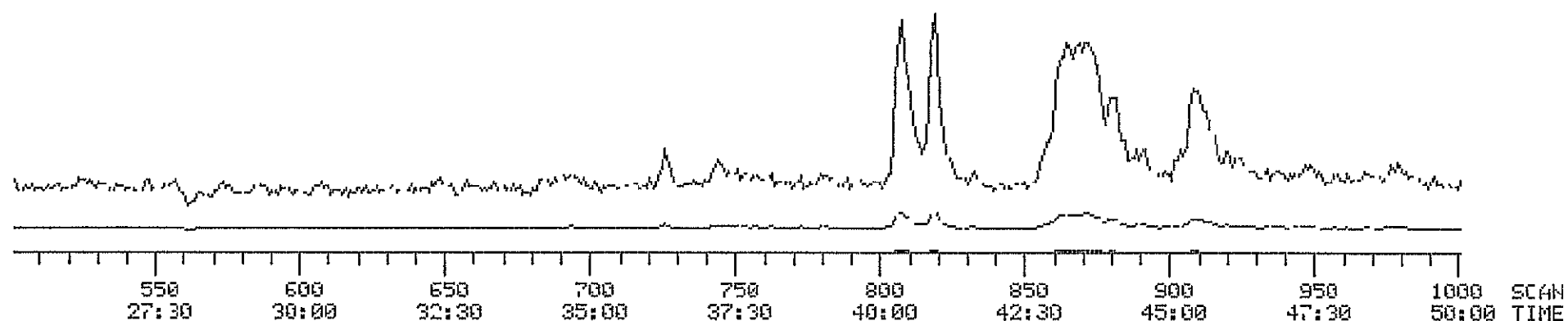
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RGC
DATE: 01 11 76 TIME: 1453
SAMPLE: TENH/13 UNTREATED

SAMPLE RUN: 1193X SCANS 501 TO 1000
CALIB. RUN: C011077PP18

Figure 13B - Untreated Product (x 10 Vertical Scale)

Expanded RGC

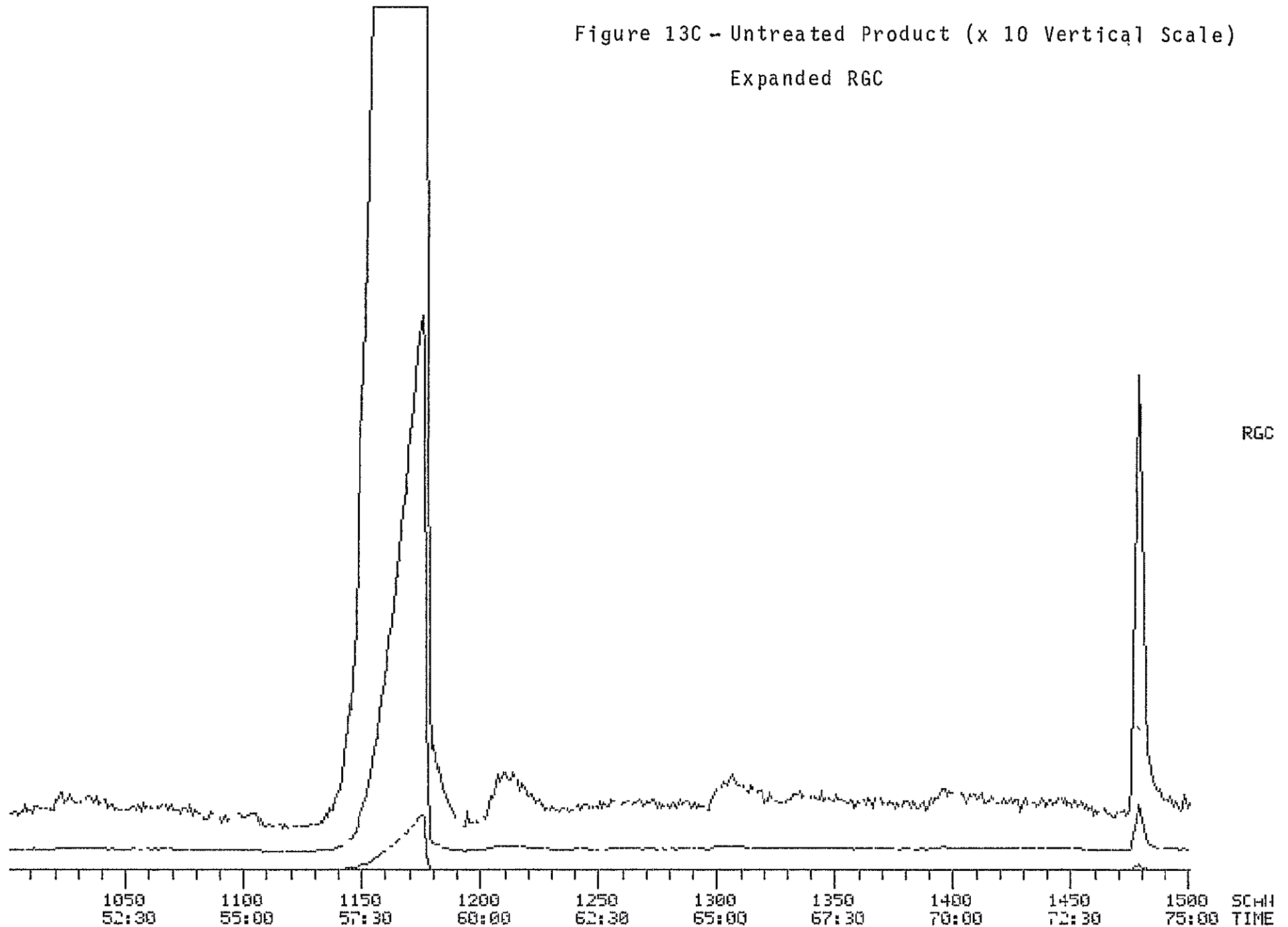


RGC
DATE: 01-11-76 TIME: 1453
SAMPLE: TEHW:13 UNTREATED

SAMPLE RUN: 1193\$ SCHN5 1001 TO 1500
CHLIB. RUN: C011077PP18

Figure 13C - Untreated Product (x 10 Vertical Scale)

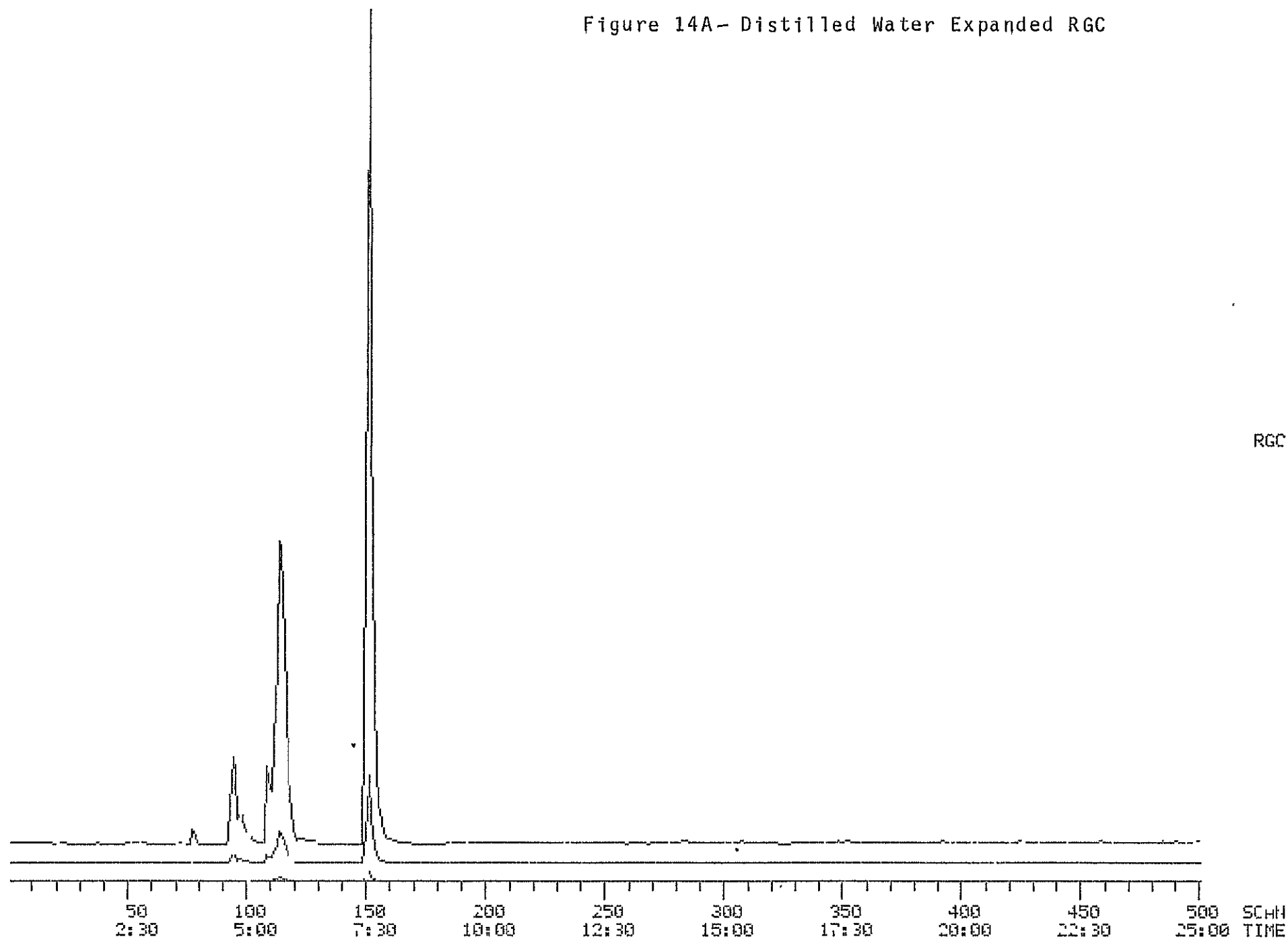
Expanded RGC



RGC
DATE: 01/13/77 TIME: 13
SAMPLE: BLANK, TENAC: 35

SAMPLE RUN: 11945 SCHIS 1 TO 500
CHLIB. RUN: C011077PP18

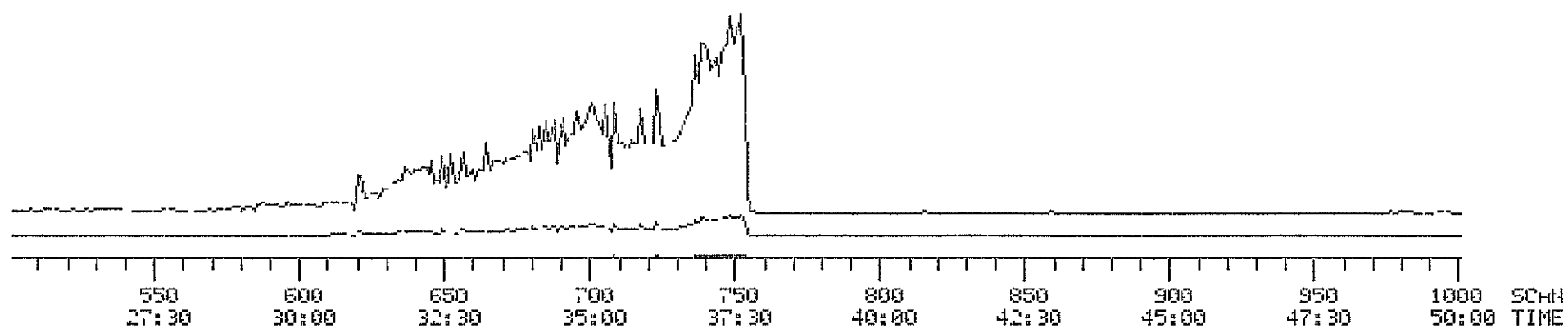
Figure 14A- Distilled Water Expanded RGC



PGC
DATE: 01 13 77 TIME: 13
SAMPLE: BLANK, TEMP: 35

SAMPLE RUN: 11943 SCHS 501 TO 1000
CHLIB. RUN: C011077PP18

Figure 14B-- Distilled Water Expanded RGC



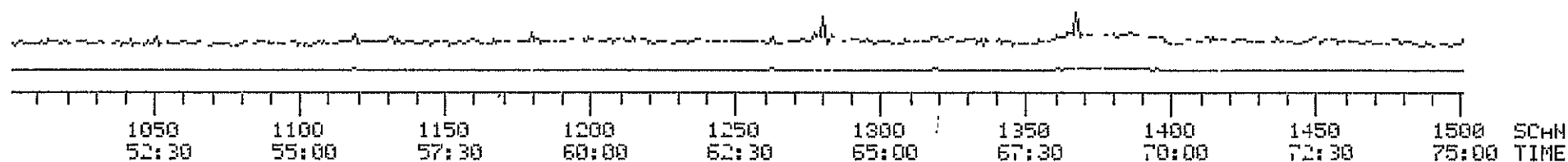
RGC
DATE: 01/13/77 TIME: 13
SAMPLE: BLANK, TEMP: 35

SAMPLE RUN: 11945 SCANS 1001 TO 1500
CALIB. RUN: C011077FP18

Figure 14C - Distilled Water (x 10 Vertical Scale)

Expanded RGC

RGC

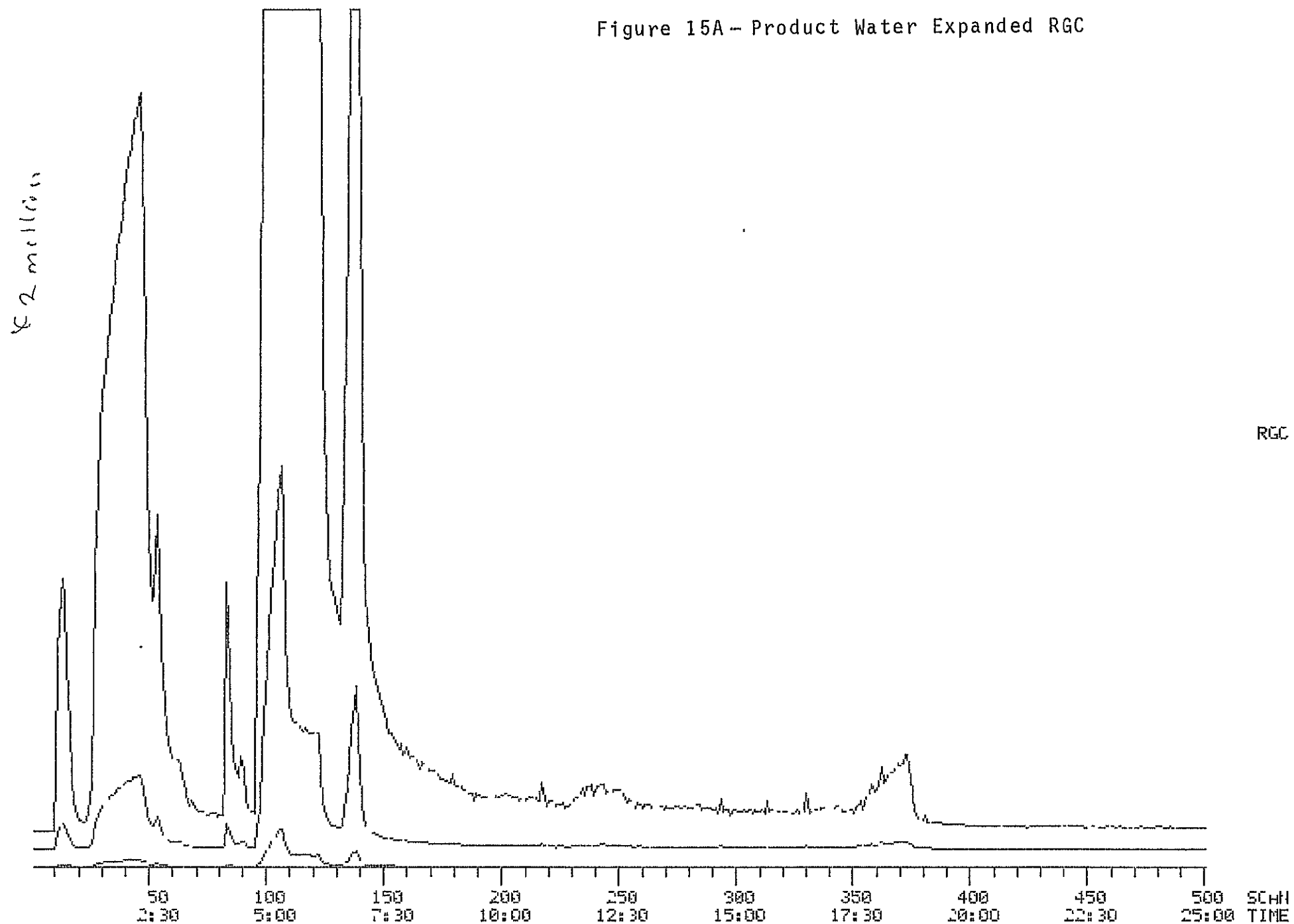


PGC
DATE: 01 11 76 TIME: 2042
SAMPLE: 100D PRODUCT

SAMPLE RUN: 11958
CHLIB. PUN: CA11077PP18

SCANS 1 TO 500

Figure 15A - Product Water Expanded RGC



PGC
DATE: 01 11/76 TIME: 2042
SAMPLE: HCO PRODUCT

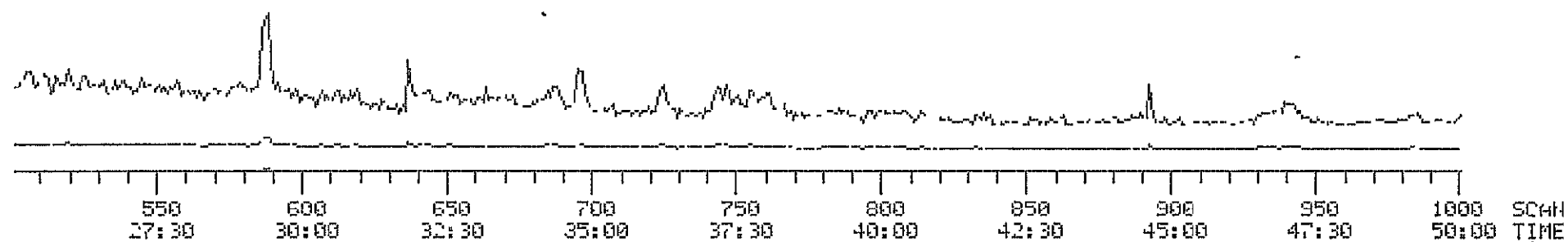
SAMPLE RUN: 11955
CHLIB. RUN: C011077PP18

SCANS 501 TO 1000

Figure 15B - Product Water (x 10 Vertical Scale)

Expanded RGC

RGC



PGC
DATE: 01-11-76 TIME: 2042
SAMPLE: HCD PRODUCT

SAMPLE RUN: 11953
CALIB. RUN: C011077PP18

SCANS 1001 TO 1450

Figure 15C -- Product Water (x 10 Vertical Scale)

Expanded RGC

X 200 X

RGC

